AMERICAN SOCIETY
for
TESTING MATERIALS

Reg. U. S. Pat. Off.

PROCEEDINGS
of the
FORTY-THIRD ANNUAL MEETING
Held at Atlantic City, N. J.
June 24–28, 1940

VOLUME 40
COMMITTEE REPORTS
TECHNICAL PAPERS

Published by the
AMERICAN SOCIETY FOR TESTING MATERIALS
260 S. Broad St., Philadelphia, Pa.

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AMERICAN SOCIETY FOR TESTING MATERIALS
Printed in Baltimore, U. S. A.
January, 1941

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SUMMARY OF PROCEEDINGS OF THE FORTY-THIRD ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 24–28, 1940

The following Summary records the action taken by the Society at the Annual Meeting on the Recommendations Affecting Standards and Tentative Standards appearing in the annual reports of the standing committees. In the individual committee reports attention is called through an editorial footnote to any modifications of the recommendations or where the recommendations of the committee were not approved. In all other instances it is to be understood that the recommendations were approved as submitted by the committee. The new and revised standards and tentative standards are published in the 1940 Supplement to the Book of A.S.T.M. Standards, Parts I, II, and III. Except as noted, the reports and papers appear in this volume.

This summary of the Forty-third Annual Meeting of the American Society for Testing Materials, held at Chalfonte-Haddon Hall, Atlantic City, N. J., June 24–28, 1940, is a record of the transactions of the meeting including the actions taken on the various recommendations submitted by the standing committees. In all, 25 sessions were held,—22 formal sessions and three round-table or topical discussions—as well as the Third A.S.T.M. Photographic Exhibition and Competition, and the Twenty-third Annual A.S.T.M. Golf Tournament.

The *Proceedings* are set forth session by session. There were 55 reports and 77 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentative standards and proposed revisions of existing standards, and as a separate group, the approval of matters that were referred to letter ballot of the Society, as for example, the adoption of tentative standards as standard or the adoption of revisions of standards. Accordingly, wherever the action is reported as "adopted as standard," it is understood that this indicates approval of the annual meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" are self evident as indicating acceptance by the Society at the annual meeting for publication as tentative.

While all of the items on the program are recorded under the particular session in which they were presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of the reports together with the page reference where the action thereon is recorded.

The registered attendance of the meeting is as follows: Members present or represented, 912; committee members, 256; guests, 273; total, 1441; ladies, 295.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, was canvassed on September 3, 1940, and all actions were approved.

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TOPICAL DISCUSSION ON RADIOGRAPHIC TESTING OF AIRPLANE COMPONENTS

Monday, June 24, 8 p.m.

Chairman: H. H. Lester.

The subject of Radiographic Testing of Airplane Components was discussed at a meeting held on Monday evening under the guidance of Committee E-7 on Radiographic Testing. Three informal papers were presented by the respective authors as follows:

Quality and Quantity X-ray Examination of Aircraft Material—T. A. Triplett

Radiography of Aluminum Castings and Forgings for Airplanes—Kent van Horn

Technique and Organization in the Radiography of Aircraft Castings—L. W. Ball

FORMAL OPENING SESSION
PRESIDENT'S ADDRESS

Tuesday, June 25, 10.30 a.m.

Session Co-Chairmen: President H. H. Morgan; Past-President A. C. Fieldner.

President H. H. Morgan in the chair.

Committee E-9 on Research:

Report presented from manuscript by T. S. Fuller, chairman.

Committee E-10 on Standards:

Report presented, in the absence of the chairman, by the Assistant Secretary.

Executive Committee:

This report, which discussed Society activities, covered membership, publications, finances, and administrative matters relating to committee activities and inter-Society relations, was presented by the Secretary-Treasurer, C. L. Warwick.

The chairman introduced Lieut. Col. William C. Young of the Planning Branch of the Office of the Assistant Secretary of War, who spoke on "Materials Standards in National Preparedness."[2] Colonel Young discussed the significance of industrial mobilization and the importance in this program of adequate materials specifications. He covered such factors as the variety of materials required, the quantities needed, and the production problems involved. He also outlined the use of educational orders as well as production studies, mentioning the careful consideration which has been given to the economic factor. The work of the Munitions Board was described and particularly the industrial mobilization plan which is a guide to be available in time of a national emergency. Colonel Young stressed the importance of the specification work of the various Government departments and also the work of the various technical societies including the A.S.T.M., citing that this organization was in an excellent position to make an important contribution in the field of standardization of materials for production of military equipment.

Past-President A. C. Fieldner then assumed the chair and introduced the President, H. H. Morgan.

Mr. Morgan then delivered the annual Presidential Address on "Standard Specifications for Materials and Their Commercial Importance." Before presenting his address, President Morgan referred briefly to the appalling events of the day which may have such far-reaching effects upon international affairs and upon conditions and living standards in this country. In the light of present-day developments he considered the subject of his address and that of Colonel Young's to be especially timely since they cover the importance and need of standardization of materials in commercial transactions and in such emergencies as may develop.

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[2] Published in ASTM BULLETIN, No. 105, August, 1940, p. 17.

In his address President Morgan discussed the importance of having, as an adjunct to a purchase order for the fundamental materials of industry, a definition of the buyers' requirements which is useful in commercial trade. He stated that a good specification provides a means by which the buyer and seller may speak the same language, thus facilitating commerce between the two, and opening the way for more specific and direct benefits. President Morgan stressed the fact that the practical importance of specifications hinges directly on their ability to assure the buyer that he is receiving exactly the goods he orders. He referred to the wide use of A.S.T.M. specifications in the extensive field of engineering construction materials as concrete evidence of their commercial importance. In concluding he stated that adequate specifications for materials with competent inspection assure to the buyer a maximum usefulness and serviceability of material at a minimum cost.

Recognition of Forty-Year Members:

The chairman announced that in accordance with the action taken by the Executive Committee, certificates were being awarded this year to four individuals and four companies who had attained 40 yr. of membership in the Society. Presentation of the certificates was then made by President Morgan to the following:

F. H. Clark, Consulting Engineer, New York, N. Y.
T. L. Condron, Consulting Structural Engineer, Chicago, Ill.
Robert Job, Vice-President, Milton Hersey Co., Ltd., Montreal, P.Q., Canada.
Charles B. Wing, Emeritus Professor of Structural Engineering, Stanford University, Palo Alto, Calif.
American Steel and Wire Co., Cleveland Ohio.
The Colorado Fuel and Iron Co., Pueblo, Colo.
National Tube Co., Pittsburgh, Pa.
John A. Roebling's Sons Co., Trenton, N. J.

Award of Honorary Memberships:

For the Award of Honorary Memberships, Past-President Fieldner called upon Past-President K. G. Mackenzie to present Past-President Cloyd M. Chapman; on Past-President H. F. Moore to present H. E. Smith; and on Past-President T. G. Delbridge to present in absentia Past-President T. R. Lawson who was unable to be present due to illness. Following each presentation, President Morgan made the Award of Honorary Membership.

Election of Officers:

The chairman then recognized J. O. Leech, chairman of the Committee of Tellers, who reported the results of the letter ballot on election of officers. Of the 1127 ballots cast, the results were as follows:

For President, to serve for 1 yr.:
W. M. Barr, 1121 votes.
For Vice-President, to serve for 2 yr.:
H. J. Ball, 1119 votes.
For Members of the Executive Committee, to serve for 2 yr.:
C. H. Fellows, 1116 votes.
R. C. Griffin, 1108 votes.
P. D. Merica, 1108 votes.
Jerome Strauss, 1113 votes.
Stanton Walker, 1113 votes.

The chairman then introduced the newly elected members of the Executive Committee, requesting that they rise as their names were called.

At the request of the chairman, Vice-President G. E. F. Lundell and Dean Harvey escorted to the chair the Vice-President-Elect, H. J. Ball, who expressed appreciation of the honor of his election and the hope that he might fulfill the duties of the office to the complete satisfaction of the membership.

The chairman then requested Past-President A. E. White and J. J. Shuman to escort to the chair the President-Elect, W. M. Barr, who, in acknowledging the honor of his election, said that he was not unmindful of the responsibility which accompanies the position but that it would be a pleasure to assume the leadership of an organization which is in such excellent position to be of service to the industry of this country and to our Government.

SECOND SESSION—RADIOGRAPHY

Tuesday, June 25, 2 p.m.

Session Chairman: P. E. McKinney.

Committee E-7 on Radiographic Testing:

Report presented from manuscript by H. H. Lester, chairman.

Papers:

The following papers were presented:

The Technique of Inspecting Wood Poles by X-ray—Myron Zucker,[3] presented by the author.

The Reduction of Secondary Radiation and of Excessive Radiographic Contrast by Filtration—Herman E. Seemann, presented by the author.

Precision Radiography—II—Gilbert E. Doan and Winston H. Sharp, presented from manuscript, in the absence of the authors, by H. H. Lester.

THIRD AND SIXTH SESSIONS—SYMPOSIUM ON TOOLS OF ANALYTICAL CHEMISTRY

Tuesday, June 25, 2 p.m.; and
Wednesday, June 26, 9.30 a.m.,
respectively

Session Co-Chairmen: Vice-President G. E. F. Lundell; G. Frederick Smith.

The Symposium on Tools of Analytical Chemistry was especially arranged to be of interest to the members of the Physical and Inorganic Chemistry Division of the American Chemical Society, who were invited to participate in the symposium. The sessions were very well attended, the papers consisting of the following:

Third Session:

Some Newer Trends in Analytical Chemistry—Beverly L. Clarke, presented from manuscript by the author.

Electronic Instruments in Analysis, Testing and Control—Ralph H. Müller.

Theory and Practice of pH Determinations and Control Using the Glass Electrode—Malcolm Dole, presented from manuscript by the author.

In the absence of Mr. Müller, due to illness, his paper was available in abstract only. In view of the additional time thus being made available, Mr. Smith introduced a discussion on perchloric acid.

Sixth Session:

Some New and Forgotten Aspects of Organic Compounds in Inorganic Analysis—Harvey Diehl, presented from manuscript by the author.

Cerate Oxidimetry—G. Frederick Smith and H. H. Willard, presented from manuscript by Mr. Willard.

Micro-Gas Analysis Methods and Their Application to Research—L. A. Wooten, presented from manuscript by the author.

The symposium was supplemented by a separate session on Spectrochemical and Microanalytical Procedures held on Wednesday afternoon at 2 o'clock (see p. 19).

FOURTH SESSION—WATER

Tuesday, June 25, 8 p.m.

Session Chairman: Vice-President W. M. Barr.

Committee D-19 on Water for Industrial Uses:

Report presented by Max Hecht, chairman, and the following actions taken:

Accepted as Tentative:

Method of Reporting Results of Analysis of Industrial Waters
Revisions of:
  Sampling Plant or Confined Waters for Industrial Uses (D 510 – 38 T)

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[3] Published in ASTM BULLETIN, No. 106, October, 1940, p. 19.

Determination of the Calcium Ion and Magnesium Ion in Industrial Waters (D 511 - 38 T)
Determination of the Chloride Ion in Industrial Waters (D 512 - 38 T)
Determination of the Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Industrial Waters (D 515 - 38 T)
Determination of the Sulfate Ion in Industrial Waters (D 516 - 38 T)

Symposium on Problems in the Classification of Natural Water Intended for Industrial Use:

The Symposium on Problems in the Classification of Natural Water Intended for Industrial Use was sponsored by Committee D-19 on Water for Industrial Uses in order to develop uniformity in reporting water analysis and to secure data from which specifications may be developed, these data permitting the expression of corrosivity and deposit-protection characteristics.

The symposium was introduced by F. N. Speller, Consulting Metallurgical Engineer, and the following papers were then presented:

Reporting the Results of Water Analysis—Robert C. Adams, presented by the author.

A Review of Data on the Relationship of Corrosivity of Water to Its Chemical Analysis—V. V. Kendall, presented from manuscript by the author.

Measuring the Scale-Forming and Corrosive Tendencies of Water by Short-Time Tests—Everett P. Partridge and G. B. Hatch, presented from manuscript by Mr. Partridge.

A Method of Measuring Corrosiveness—J. H. Walker, presented by the author.

FIFTH SESSION—METHODS OF TESTING
Tuesday, June 25, 8 p.m.

Session Chairman: J. R. Townsend.

Committee E-1 on Methods of Testing:

Report presented by W. H. Fulweiler, chairman, and the following actions taken:

Accepted as Tentative:

Methods of Tension Testing of Metallic Materials (E 8 - 40 T), to replace immediately Standard Methods E 8 - 36
Recommended Practices for Designation of Numerical Requirements in Standards
Revision of Definitions of Terms Relating to Rheological Properties of Matter (E 24 - 37 T)
Revision of Spec. for A.S.T.M. Thermometers (E 1 - 39 T), revised requirements for turpentine distillation thermometer E 1 (27 C - 39)

Mr. Fulweiler then called upon H. F. Moore to describe the activities of Technical Committee I on Mechanical Testing; on E. C. Bingham to report on the work of Technical Committee II on Consistency, Plasticity, and Related Properties; and on L. T. Work to summarize the activities of Technical Committee III on Particle Size and Shape.

Committee E-3 on Chemical Analysis of Metals:

Report presented from manuscript by G. E. F. Lundell, chairman.

Committee E-2 on Spectrographic Analysis:

Report presented from manuscript by H. V. Churchill, chairman.

Committee E-8 on Nomenclature and Definitions:

Report presented by Cloyd M. Chapman, chairman.

Papers:

The following papers were presented:

An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals—Paul G. Jones and H. F. Moore, presented by Mr. Jones.

Speed in Tension Testing and Its Influence on Yield Point Values—Lawford H. Fry, presented from manuscript by the author.

Quantitative Spectrographic Analysis of Steels—S. Vigo,[4] presented from manuscript by the author.

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[4] Published in ASTM BULLETIN, No. 107, December, 1940, p. 17.

SEVENTH SESSION—SYMPOSIUM ON SIGNIFICANCE OF THE TENSION TEST OF METALS IN RELATION TO DESIGN

Wednesday, June 26, 9.30 a.m.

Session Chairman: John M. Lessells.

The Symposium on Significance of the Tension Test of Metals in Relation to Design was arranged in view of the differences of opinion regarding the significance of the results obtained. It was sponsored in cooperation with the American Institute of Physics and the Applied Mechanics Division of The American Society of Mechanical Engineers. The Symposium was introduced by John M. Lessells, who then called upon the following who had prepared formal discussions covering several features of the subject:

C. W. MacGregor—The Tension Test.

F. B. Seely—The Strength Features of the Tension Test.

H. W. Gillett—The Limited Significance of the Ductility Features of the Tension Test.

The meeting was then thrown open to general discussion, in which a number of those present participated.

EIGHTH SESSION—CERAMIC AND MASONRY MATERIALS

Wednesday, June 26, 2 p.m.

Session Chairman: A. T. Goldbeck.

Committee C-15 on Manufactured Masonry Units:

Report presented by D. E. Parsons chairman, and the following actions taken:

Accepted as Tentative:

- Revision of Spec. for Building Brick (Made from Clay or Shale) (C 62 – 39 T)

Adopted as Standard:

- Spec. for Solid Load-Bearing Concrete Masonry Units (C 145 – 39 T)

Committee C-8 on Refractories:

Report presented, in the absence of the chairman, by G. A. Downsbrough, and the following actions taken:

Accepted as Tentative:

- Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (C 63 – 40 T), to replace the present Spec. C 63 – 39
- Spec. for Refractories for Heavy Duty Stationary Boiler Service, and Spec. for Refractories for Moderate Duty Stationary Boiler Service, to replace the present Spec. C 64 – 39
- Spec. for Refractories for Incinerators (C 106 – 40 T), to replace the present Spec. C 106 – 39
- Test for Warpage of Refractory Brick and Tile
- Classification of Insulating Fire Brick
- Revisions of:
  - Definitions of Terms Relating to Refractories (C 71 – 39 T)
  - Spec. for Ground Fire Clay (C 105 – 36)
  - Chemical Analysis of Refractory Materials (C 18 – 39)
  - Definitions of Fireclay and Alumina-Diaspore Refractories (C 27 – 39)

Adopted as Standard:

- Test for True Specific Gravity of Burned Refractory Materials (C 135 – 38 T)
- Revision of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 36)
- Revision of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 37)

Withdrawal of Standards:

- Spec. for Fireclay Brick for Malleable Furnaces with Removable Bungs and for Annealing Ovens (C 63 – 39)
- Spec. for Fireclay Brick for Stationary Boiler Service (C 64 – 39)
- Spec. for Refractories for Construction of Incinerators (C 106 – 39)

Editorial Change Accepted:

- Test for Size and Bulk Density of Refractory Brick (C 134 – 39 T)

Committee C-4 on Clay Pipe:

Report presented, in the absence of the chairman, by J. C. Riedel, vice-chairman, and the following actions taken:

Adopted as Standard:

Revision of Spec. for Clay Sewer Pipe (C 13 - 35)

Withdrawal of Tentative Standard:

Definitions of Terms Relating to Clay Sewer Pipe (C 8 - 35 T)

Committee C-5 on Fire Tests of Materials and Construction:

Report presented by R. P. Miller, chairman, and the following actions taken:

Accepted as Tentative:

Fire Tests of Door Assemblies

Revision of Spec. for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 - 37 T)

Revision of Spec. for Fire Tests of Building Construction and Materials (C 19 - 33), with minor additional changes as follows:

Section 3 (a).—Change the last part of the first sentence from its form as preprinted: namely,

> or in the case of base metal thermocouples, enclosed in sealed standard-weight ½-in. wrought steel pipe.

to read as follows:

> or as an alternative in the case of base metal thermocouples, enclosed in sealed standard weight ½-in. black wrought steel or black wrought iron pipe.

Section 22.—In Paragraphs (*a*), (*b*) and (*c*), change the word "carrying" to read "having."

Paper:

The following paper was presented:

Relation of Water Absorption and Strength of Brick to Abrasive Resistance—J. W. McBurney, R. H. Brink, and A. R. Eberle, presented by Mr. McBurney.

NINTH SESSION—STEEL, FERRO-ALLOYS, METALLOGRAPHY

Wednesday, June 26, 2 p.m.

Session Chairman: J. O. Leech.

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Carbon-Steel Forgings for General Industrial Use

Spec. for Carbon-Steel Forgings for Locomotives and Cars

Spec. for Alloy-Steel Forgings for Locomotives and Cars

Spec. for Alloy-Steel Forgings for General Industrial Use.

In the above specifications the following changes, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[5]

In Section 10 (*e*) of each of the above four specifications add the word "reading" after the word "extensometer."

In the Specifications for Carbon-Steel Forgings for General Industrial Use add a new class designated C1, covering annealed or normalized mild steel forgings for bridges. Insert in Table I the following requirements as to physical properties: For all sizes a minimum tensile strength of 66,000 psi. and a minimum yield point of 33,000 psi. For forgings 12 in. and under in outside diameter or over-all thickness a minimum elongation in 2 in. of 23 per cent, and a minimum reduction of area of 36 per cent. For forgings over 12 to 20 in., incl., a minimum elongation and reduction of area of 22 per cent and 34 per cent, respectively.

Various editorial changes were approved in the four specifications listed, including rearrangement of the tables of physical properties. The photomicrographs in the Specifications for Carbon-Steel and for Alloy-Steel Forgings for Locomotives will be corrected.

Spec. for Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings, with the following additional changes subject to favorable letter ballot of Committee A-1:[6]

Section 2 (a).—In the second sentence, change the word "or" to read "and."

Section 4 (b).—Delete the phrase "and the chemical analyses of finished fittings."

Section 7.—Change from its present form to read as follows by the addition of the italicized words and the omission of those in brackets:

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[5] Committee A-1 reported the following results of the letter ballot vote of a total of 164 ballots returned from a voting membership of 229: Carbon-Steel Forgings for General Industrial Use, affirmative 106, negative 1; Carbon-Steel Forgings for Locomotives and Cars, affirmative 90, negative 0; Alloy-Steel Forgings for Locomotives and Cars, affirmative 87, negative 0; Alloy-Steel Forgings for General Industrial Use, affirmative 100, negative 1.

[6] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 164 members returned their ballots, of whom 66 voted affirmatively, 4 negatively.

7. Tension tests of the finished fittings are not required. [Tensile properties] *Tension testing* of fittings *when required* shall be a matter of agreement between the manufacturer and the purchaser.

Section 8 (*a*).—Change from the form as preprinted by incorporating the method of determining the test pressure, the revised section to read as follows:

8. (*a*) Welding fittings shall be capable of withstanding a hydrostatic pressure test determined by the formula

$$P = \frac{2St}{D}$$

where:

P = maximum hydrostatic test pressure, in pounds per square inch,

S = 0.50 times the minimum specified yield point of the designated material, in pounds per square inch,

t = nominal wall thickness, in inches, of designated schedule number,

D = outside diameter at bevel, in inches.

Section 11 (*b*).—Change the first sentence from its present form to read as follows by the addition of the italicized words:

(*b*) On all alloy-steel fittings, the manufacturer's name or trade-mark and the designation of the material specification shall be marked with a steel stamp *or by any other suitable method agreed upon by the manufacturer and the purchaser* so that the marking will be of a permanent nature.

In Table I, editorial corrections will be incorporated, and a footnote will be added indicating that reference to designations of other A.S.T.M. specifications is made only for cross reference to the respective chemical compositions.

Revisions of:

Spec. for Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 – 39 T)

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Super-Heater Tubes (A 209 – 38 T)

Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 – 39 T)

Spec. for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 – 39 T)

The revised marking clauses of Specifications A 216 and A 217 were further modified so that the marking of the manufacturer's name or trade mark and the word "Steel" will not be required.

Spec. for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 38 T); with the following additional change, not preprinted, approved by unanimous consent, subject to favorable letter ballot of Committee A-1:[7]

Section 19.—Add a new second sentence to Paragraph (*a*) to read as follows:

At the discretion of the inspector representing the purchaser, finished pipe shall be subject to rejection if surface defects acceptable under Paragraph (*c*) are not scattered, but appear over a large area in excess of what is considered "workmanlike finish."

Add a new first sentence to Paragraph (*b*) to read as follows:

All defects shall be explored for depth.

Delete Paragraph (*c*), and substitute the following:

(*c*) *Machining or Grinding Defects.*—Surface defects shall be classified and treated as follows:

(*1*) U-bottom mechanical marks and abrasions[a] excepting pits, tears, scabs, slivers, etc., shall be acceptable without grinding or machining, if not deeper than 12.5 per cent of the nominal wall thickness nor in any case deeper than $\frac{1}{16}$ in. All V-bottom marks and abrasions and U-bottom marks and abrasions deeper than $\frac{1}{16}$ in. but not deeper than 12.5 per cent of the nominal wall thickness shall be removed by grinding or machining to sound metal. Where the width of any mark or abrasion is less than the depth it shall be classified as V-bottomed.

(*2*) Pipe showing inside or outside surface checks (fish scale) $\frac{1}{64}$ in. or less in depth need not have these defects removed. Such defects over $\frac{1}{64}$ in. but not more than $\frac{1}{32}$ in. in depth shall be removed by machining or grinding. Pipe on which these defects are more than $\frac{1}{32}$ in. in depth shall be rejected, unless the manufacturer can demonstrate to the purchaser that the defects are not injurious as defined in Paragraph (*b*).

(*3*) Pipe showing scabs, seams, laps, tears, or slivers not deeper than 5 per cent of the nominal wall thickness need not have these defects removed. If deeper than 5 per cent such defects shall be removed by machining or grinding providing they are not injurious as defined in Paragraph (*b*).

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[a] Marks and abrasions are defined as cable marks, dinges, guide marks, roll marks, ball scratches, scores, etc.

[7] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 164 members returned their ballots, of whom 74 voted affirmatively, 0 negatively.

(*4*) Pits which do not fall under the classification of injurious defects as defined in Paragraph (*b*) need not be removed.

(*5*) When grinding or machining is permitted, the outside diameter at the point of grinding or machining may be reduced by the amount so removed. Should it be impracticable to secure a direct measurement, the wall thickness at the point of grinding, or at a defect not required to be removed, shall be determined by deducting the amount removed in grinding, or the depth of the defect, from the minimum measured wall thickness at the ends of the pipe, and the remainder shall not be less than 87.5 per cent of the nominal wall thickness.

(*6*) Machining or grinding shall follow the inspection at the mill of the pipe as rolled and shall be followed by supplementary visual inspection.

Supplementary Requirements.—In Section S5 delete the first sentence.

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 39 T), with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[8]

Section 20 (*a*), (*b*) *and* (*c*).—Make the same changes in this section as recommended above in Section 19 (*a*), (*b*) and (*c*) of Specifications A 158 - 38 T.

Supplementary Requirements.—Change Paragraph S5 to read as follows:

S5. The steel shall have a homogeneous structure as shown by the etching tests described in Paragraph S6. The grain size of the steel shall conform to the requirements of Section 2 (*d*) except that in nominal pipe sizes 3 in. and over made to Schedules 140 and 160, each piece of pipe in the as-finished condition shall show a structural grain size[a] corresponding to an A.S.T.M. grain size of 3 to 6 in accordance with the Tentative Classification of Austenite Grain Size in Steels (A.S.T.M. Designation: E 19) of the American Society for Testing Materials.

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[a] The term "structural grain size" refers to the predominant grain as evidenced by ferrite grains, Widmänstatten areas, or pearlite patches.

Spec. for Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 - 39), Spec. for Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 - 36) and Spec. for Alloy-Steel Castings for Structural Purposes (A 148 - 36), with the following change in each, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[9]

Section 8.—Change Paragraph (*c*) in Spec. A 27 and A 87 and Paragraph (*b*) in Spec. A 148 from their present forms to read as follows:

The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine, or by the use of dividers, or by an extensometer, at a crosshead speed not to exceed $\frac{1}{8}$ in. per min. Where a definite yield point is not exhibited, the yield strength corresponding to a limiting permanent offset of 0.2 per cent of the gage length of the specimen shall be used instead. The "offset method" of determining yield strength as described in the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials shall be followed. The tensile strength shall be determined at a speed of head not to exceed $1\frac{1}{2}$ in. per min.

While the same change was approved in the Tentative Specifications for Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 - 39 T), the committee did not wish the tentative specifications to be taken from the current Book of Standards and appear in the 1940 Supplement and is therefore withholding recommending the change to the Society.

Spec. for Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures (A 193 - 39 T), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[10]

Table I.—Change the molybdenum content for grade B5 from "0.45 to 0.65 per cent" to read "0.40 to 0.60 per cent"; and change the silicon content for grades B7 and B7a from "0.15 to 0.25 per cent" to read "0.15 to 0.30 per cent."

Spec. for Structural Steel for Locomotives and Cars (A 113 - 39)

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[8] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 164 members returned their ballots, of whom 72 voted affirmatively, 2 negatively.

[9] Committee A-1 reported the following results of the letter ballot vote of a total of 147 ballots returned from a voting membership of 229: A 27, affirmative 93, negative 0; A 87, affirmative 93, negative 0; A 148, affirmative 93, negative 0.

[10] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 147 members returned their ballots, of whom 85 voted affirmatively, 0 negatively.

Spec. for Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 – 36) and Spec. for Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 37), this recommendation representing a change in the report as preprinted which had proposed the revisions in these two standards for immediate adoption.

Adopted as Standard:

Spec. for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 38 T), as revised

Spec. for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 38 T), as revised

Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 39 T), as revised

Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 39 T), as revised

Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 39 T), with the following additional change, not preprinted: The manganese content of grade T20 will be changed from "0.60 to 1.50 per cent" to read "3.00 per cent, max."

Spec. for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 38 T) with the following change, not preprinted:[11, 12]

Section 5.—At the end of this section add a new Paragraph (*c*) to read as follows:

(*c*) Superficial ruptures as a result of minor surface imperfections shall not be cause for rejection.

The recommendations to adopt the six tentative specifications listed above with the revisions published in the report and two additional changes itemized above were not included in the report as preprinted but were accepted by unanimous consent for reference to Society letter ballot subject to favorable letter ballot of Committee A-1.[12]

Spec. for Spiral-Welded Steel or Iron Pipe (A 211 – 38 T), as revised.

Revisions of:

Spec. for Boiler Rivet Steel and Rivets (A 31 – 39)

Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 36)

Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 37)

Spec. for Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 – 38)

Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 39)

Spec. for Commercial Quality Hot-Rolled Bar Steels (A 107 – 39)

Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for Service at Temperatures from 750 to 1100 F. (A 182 – 39)

Spec. for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 – 36)

Spec. for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 39)

The revised marking clauses of Specifications A 95 and A 157 were further modified so that the marking of the manufacturer's name or trade mark and the word "Steel" will not be required.

Spec. for Steel Plates of Structural Quality for Forge Welding (A 78 – 39), with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[13]

Title.—Change from its present form, to read as follows:

Specifications for Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding

Section 1.—Change from its present form, to read as follows:

1. These specifications cover two grades of carbon-steel plates of structural quality suitable for welding. They are suitable for forge welding when the requirements specified in Section 3 (*b*) are applied. The purchaser shall specify when the material is to be used for forge welding.

NOTE.—The term "forge welding" as used

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[11] These specifications, under the joint jurisdiction of Committee A-1 and Committee A-2, were referred to letter ballot of both standing committees and approved.

[12] Committee A-1 reported the following results of the letter ballot vote of a total of 147 ballots returned from a voting membership of 229: A 192 and A 213, affirmative 79, negative 0; A 210 and A 83, affirmative 82, negative 0; A 214, affirmative 77, negative 0; A 226, affirmative 76, negative 0. Committee A-2 reported the following results of the letter ballot vote on Specifications A 83: Of a total voting membership of 54, 44 members returned their ballots, of whom 39 voted affirmatively, 0 negatively.

[13] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 147 members returned their ballots, of whom 87 voted affirmatively, 4 negatively.

in these specifications refers to the process of heating with water gas, and hammering mechanically, without the use of fluxes.

Section 3 (*b*).—Change from its present form, to read as follows:

(*b*) When these specifications are used for material that is to be forge welded, the quantity of either nickel or chromium shall not exceed 0.05 per cent and, unless otherwise specified, the silicon shall not exceed 0.05 per cent.

Delete the note following this Section.

Spec. for Seamless Alloy-Steel (4 to 6 per cent Chromium) Still Tubes for Refinery Service (A 188 – 38) and Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 39), with the following additional changes, to provide for tolerances of upset ends of still tubes and clarify repairing by grinding, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[14]

Table III.—Change the phrase "length of upset, in." to read "specified length of upset, in." with a footnote indicating that "specified length of full upset is minimum."

Change the note under the title of Table III to read:

For tubes that are upset at the ends, a "go" plug gage with a diameter determined by the appropriate formula given in the table shall enter each upset end the full length of the upset. A "no-go" plug gage using the same formula without the clearance allowance shall not enter the tube.

Section 15.—Change Paragraph (*d*) to read as follows by the addition of the italicized words:

(*d*) Permissible variations *in outside diameter* for hot-rolled tubes shall apply to the upset ends of both hot-rolled and cold-drawn tubes.

Add new Paragraphs (*e*) and (*f*) to read as follows:

(*e*) For upsets that increase the wall thickness more than $\frac{1}{16}$ in., the length at any point shall not be more than 50 per cent over that specified, or a minimum of $1\frac{1}{2}$ in. For upsets that increase the wall thickness $\frac{1}{16}$ in. or less, the length at any point shall not be more than $2\frac{1}{2}$ in. over that specified.

(*f*) For upset ends of tubes, the wall thickness measured within $\frac{3}{4}$ in. from the finished end shall not exceed the actual average wall of that section by more than plus or minus 10 per cent. The actual average wall is defined as the average of the thickest and thinnest wall in that section.

Section 16 (*a*).—Change the third sentence from its present form to read as follows by the addition of the italicized words:

Minor defects may be removed by grinding, provided *that a smooth curved surface is maintained*; *that* the wall thickness is not decreased to less than that permitted in Section 15, *and such grinding shall not be limited by the permissible variations in outside diameter prescribed in Table I, except that for a distance 6 in. from each end of a tube the permissible variations in outside diameter shall be maintained.*

Specs. for Seamless Low-Carbon Steel Still Tubes for Refinery Service (A 161 – 37), with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Table III.—Make the same changes in this table as recommended above in Specifications A 188 – 38.

Section 9 (*a*).—For cold-drawn carbon-molybdenum tubes add a requirement for Brinell hardness number of "137, max."

Section 14.—Make the same change in Paragraph (*d*) as recommended above in Section 15 (*d*) of Specifications A 188 – 38. Add new Paragraphs (*e*) and (*f*) as recommended above in Specifications A 188 – 38.

Section 15 (*a*).—Make the same change in this section as recommended above in Section 16 (*a*) of Specifications A 188 – 38.

Spec. for Forged or Rolled Steel Pipe Flanges for High-Temperature Service (A 105 – 39), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[16]

Title.—Change from its present form to read as follows:

Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service.

Throughout the specifications, change the word "class" to read "grade."

Section 1 (*a*).—Change from its present form to read as follows:

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[14] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 164 members returned their ballots, of whom 76 voted affirmatively, 1 negatively.

[15] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 147 members returned their ballots, of whom 80 voted affirmatively, 1 negatively.

[16] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 147 members returned their ballots, of whom 74 voted affirmatively, 0 negatively.

(*a*) These specifications cover forged or rolled steel pipe flanges, forged fittings and valves and parts for high-temperature service (Note). The term "forgings" used in these specifications shall be understood to cover one or all of the products mentioned above, either forged or rolled.

Section 6.—Under the table of chemical requirements, change the footnote from its present form to read as follows:

[a] When flanges will be subject to fusion welding, the carbon content shall not exceed 0.35 per cent. When the carbon is restricted to 0.35 per cent max., it may be necessary to add silicon to the composition for grade II and for the heavier thicknesses of grade I flanges in order to meet the required tensile properties. The silicon content shall not exceed 0.30 per cent.

Section 10 (*c*).—Change from its present form to read as follows:

(*c*) For the purpose of making tension tests, the forging manufacturer shall supply suitable test specimens from the flange or, at his option, furnish test blanks prepared from the billets or forging bar entering into the finished product, provided such test blank has undergone approximately the same working and heat treatment as the finished product.

Spec. for Welded and Seamless Steel Pipe (A 53 - 36), with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[17]

Table I.—Change the yield point requirement for grade A pipe from "26,500 psi." to read "30,000 psi."

Section 17.—In the new marking clause, delete the word "butt-welded" and the phrase "STD for standard weight."

Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 37), the recommendation to adopt as standard the tentative revision[18] published since August, 1939, involving the use of the decimal system instead of B.w.g. was not preprinted, but was accepted with unanimous consent, subject to favorable letter ballot of Committee A-1.[19]

Spec. for Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 - 39), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[20]

Table I.—Change the requirement for carbon for class 3 from "0.20 per cent, min." to read "0.35 per cent, max." and for molybdenum from "0.45 to 0.65 per cent" to read "0.40 to 0.60 per cent."

Section 10.—Change the last sentence to read as follows by the addition of the italicized words:

The threads in the nut shall not strip when subjected to a stress equal to *130,000 psi. for class 1 and* 150,000 psi. *for classes 2, 2H, 3 and 4* figured from the mean diameter of the bolt.

Spec. for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 39), with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[21]

Table I.—For grade A pipe, insert a requirement for carbon of "0.25 per cent, max."

Sections 16 and S5.—Make the same changes in Sections 16 (*a*), (*b*) and (*c*) and S5 as recommended above in Sections 19 (*a*), (*b*) and (*c*) and S5 of Specifications A 158 - 38 T.

Withdrawal of Standards and Tentative Standards:

Spec. for Carbon-Steel and Alloy-Steel Forgings (A 18 - 30)

Spec. for Normalized Quenched-and-Tempered Alloy-Steel Forgings (A 63 - 39)

Spec. for Normalized and Tempered Alloy-Steel Forgings for Locomotives (A 133 - 33 T)

Spec. for Carbon-Steel Forgings (A 18 - 39 T)

Committee A-6 on Magnetic Properties:

Report presented by Thomas Spooner, chairman, and accepted with one typographic correction: namely, in the revised formula in Section 4 (*c*) of the Tentative Methods of Test for Measuring Interlamination Resistance and

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[17] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 147 members returned their ballots, of whom 84 voted affirmatively, 0 negatively.

[18] 1939 Book of A.S.T.M. Standards, Part I, p. 1273.

[19] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 147 members returned their ballots, of whom 80 voted affirmatively, 0 negatively.

[20] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 147 members returned their ballots, of whom 87 voted affirmatively, 0 negatively.

[21] Committee A-1 reported results of the letter ballot vote as follows: Of a total voting membership of 229, 147 members returned their ballots, of whom 75 voted affirmatively, 2 negatively.

Lamination Factor of Iron and Steel (A 34 – 39 T) the figure "18.75*R*" should read "1.875*R*."

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report presented by Jerome Strauss, chairman. The committee requested permission to publish as an appendix to its report data from tests of two stainless steel sections obtained as a result of windstorm damage to a large New York City building which had previously been inspected as part of the committee's work.

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman.

Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing:

Report presented by H. H. Morgan, chairman.

TENTH SESSION—MARBURG LECTURE AND DUDLEY MEDAL AWARD

Wednesday, June 26, 4 p.m.

Session Chairman: President H. H. Morgan.

The President, in opening the session devoted to the Edgar Marburg Lecture, referred to the double significance of the lecture instituted in 1926 to commemorate the name of the first Secretary-Treasurer of the Society, Dr. Edgar Marburg, and to bring to the membership valuable expositions on notable developments in engineering and the sciences.

The President then introduced the fifteenth Edgar Marburg Lecturer, P. H. Bates, Chief, Clay and Silicate Products Division, National Bureau of Standards, who had selected as the subject of the lecture "Portland Cement—Theories (Proved and Otherwise) and Specifications." Before delivering the lecture, Mr. Bates briefly recalled his experience as an undergraduate when he studied under Professor Marburg at the University of Pennsylvania. He also referred to his contact with Dr. Charles B. Dudley, the first President of the Society, with whom he worked in the Pennsylvania Railroad Laboratory at Altoona, Pa. Mr. Bates considered it a privilege to have had the honor of being associated with these two charter members of the Society. He paid an especial tribute to Professor Marburg as a great teacher and a capable organizer and executive.

In his lecture, Mr. Bates discussed the difficulty of defining cement in the ordinary dictionary manner. He covered briefly the history of the material and discussed in some detail the question "What is Portland Cement?" referring to the various constituents such as tricalcium silicate and the other major constituents. He reviewed the reactions of cement with water and particularly commented on the new specifications for cement in which discussion he covered the question of whether five types were needed.

The rapid change in viewpoints on the use of admixtures was cited and he referred to many studies that are under way on the effects of various materials. He indicated that there seems to be no brighter prospect of improving cements as they are now produced other than through the use of admixtures. Mr. Bates referred to five interesting tests developed in recent years for use in accepting cement, stating that the trial of these methods should be done in an open-minded manner.

Throughout the lecture, Mr. Bates stressed the need of additional research on many problems. In conclusion he referred to the fact that while there are still many uncertainties regarding the true nature of portland cement, such as its reaction with water, and how it will deport itself under certain trying conditions, this in no way detracts from the proven findings of its tremendous use and the fact that the results are startlingly satisfactory.

President Morgan in expressing appreciation to Mr. Bates for his fine exposition on a subject of particular interest to the A.S.T.M., presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

President Morgan then introduced E. E. Thum, Chairman of the Dudley Medal Committee, who presented as the recipients of the fourteenth award of the Charles B. Dudley Medal Messrs. T. F. Willis and M. E. De Reus, Research Engineer and Junior Engineer, respectively, Bureau of Materials, Missouri State Highway Dept., for their paper on "Thermal Volume Change and Elasticity of Aggregates and Their Effect on Concrete," presented at the 1939 annual meeting of the Society. The medal is awarded annually by the Society to the author or authors of a paper of outstanding merit constituting an original contribution on research in engineering materials. Mr. Thum stated that the committee believed that the authors of this paper eminently merit the recognition conveyed by the Dudley Medal, since the paper represents an original research on the properties of concrete aggregates that had developed some entirely new information on an important material of construction and that by the use of ingenious and simple test equipment described, it should stimulate further work in this field.

On behalf of and as the representative of the Society, President Morgan then made the fourteenth award of the Charles B. Dudley Medal to Messrs. T. F. Willis and M. E. De Reus.

In accepting the medal on behalf of the recipients, Mr. Willis expressed deep appreciation of the honor accorded by the award of the Dudley Medal and stated that the receiving of it would serve as a very pleasant stimulus to their further work.

ELEVENTH SESSION—CORROSION, IRON

Wednesday, June 26, 8 p.m.

Session Co-Chairmen: Sam Tour; H. A. Schwartz.

Committee A-5 on Corrosion of Iron and Steel:

Report presented by W. H. Finkeldey, chairman, and the following actions taken:

Accepted as Tentative:

Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles
Revisions of:
  Spec. for Electrodeposited Coatings of Zinc on Steel (A 164 - 39 T)
  Spec. for Electrodeposited Coatings of Cadmium on Steel (A 165 - 39 T)
  Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 39 T)

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented by T. S. Fuller, chairman.

Mr. Fuller then presented Mr. L. J. Gorman who introduced the authors of the following two papers appended to the report:

Some Observations of the Potentials of Metals and Alloys in Sea Water—F. L. LaQue and G. L. Cox, presented by Mr. LaQue.

The Controlling Factors in Galvanic Corrosion—W. A. Wesley, presented by the author.

Joint Committee on Exposure Tests of Plating on the Non-Ferrous Metals:

Informal progress report presented by William Blum, chairman.

Committee A-3 on Cast Iron:

Report presented in the absence of the chairman, by J. T. MacKenzie, and the following actions taken:

Adopted as Standard:

Spec. for Automotive Gray-Iron Castings (A 159 - 35 T)
Spec. for Lightweight and Thin-Sectioned Gray-Iron Castings (A 190 - 36 T)
Revision of Spec. for Gray-Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 - 30)

Sectional Committee A21 on Specifications for Cast-Iron Pipe and Fittings:

Informal progress report presented, in the absence of the chairman, by J. T. MacKenzie.

Committee A-7 on Malleable Iron Castings:

Report presented, in the absence of the chairman, by F. L. Wolf, vice-chairman.

Papers:

The following papers were presented:

Atmospheric Durability of Steels Containing Nickel and Copper—N. B. Pilling and W. A. Wesley, presented by Mr. Pilling.

Mechanical Properties of Gray Cast Iron—Jasper O. Draffin, W. Leighton Collins and Carl H. Casberg, presented from manuscript by Mr. Collins.

Properties of Commercial Pearlitic Malleable Iron—C. H. Lorig,[22] presented by the author.

Stress-Strain Relations for Malleable Cast Iron in Tension with Special Attention to Yield Point Determination—R. D. Landon, presented by the author.

TWELFTH SESSION—CEMENT, LIME, GYPSUM, CONCRETE

Wednesday, June 26, 8 p.m.

Session Co-Chairmen: President H. H. Morgan; M. A. Swayze.

Committee C-11 on Gypsum:

Report presented, in the absence of the chairman, by the Secretary-Treasurer.

Accepted as Tentative:

Revision of Definitions of Terms Relating to Gypsum (C 11 - 31 T)
Revision of Spec. for Gypsum (C 22 - 25)

Adopted as Standard:

Revisions of:
  Spec. for Gypsum Plasters (C 28 - 39)
  Spec. for Gypsum Molding Plaster (C 59 - 30)
  Spec. for Gypsum Pottery Plaster (C 60 - 30)
  Spec. for Keene's Cement (C 61 - 30)
  Spec. for Calcined Gypsum for Dental Plasters (C 72 - 30)
  Spec. for Gypsum Lath (C 37 - 34)
  Testing Gypsum and Gypsum Products (C 26 - 39)

Report of Committee C-7 on Lime:

Report presented by J. R. Withrow, chairman, and the following action taken:

Accepted as Tentative:

Revision of Spec. for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 - 24); not having been preprinted was accepted by unanimous consent subject to favorable letter ballot of Committee C-7:[23]

Section 1.—Change the note following this section from its present form to read as follows:

NOTE.—In the manufacture of silica brick, silica in the form of massive quartzite or quartz conglomerate is ground until the particles are less than $\frac{1}{4}$ in. in size. Lime either in the form of slaked lime or hydrated lime is then added in quantities varying from 1.5 to 3.0 per cent CaO and the shapes are molded and dried. They are then burned in down-draft or tunnel kilns until most of the quartzite has been converted into tridymite or cristobalite.

Section 3.—In the table of chemical composition change the requirement for calcium oxide from "92.0 per cent" to read "90 per cent," and increase the permissible magnesium oxide content from "3.0 per cent" to read "4.5 per cent."

Committee C-12 on Mortars for Unit Masonry:

Report presented by J. W. McBurney, chairman.

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[22] Published in ASTM BULLETIN, No. 105, August 1940, p. 29.

[23] Committee C-7 reported results of the letter ballot vote as follows: Of a total voting membership of 50, 30 members returned their ballots, of whom 18 voted affirmatively, 0 negatively.

Sectional Committee A42 on Specifications for Plastering:

Informal report presented, in the absence of the chairman, by J. W. McBurney, secretary.

Committee C-1 on Cement:

Report presented by P. H. Bates, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Portland Cement, to become effective September 2, 1940

Test for Autoclave Expansion of Portland Cement

Revisions of Methods of Chemical Analysis of Portland Cement (C 114 - 39 T), comprising new quick procedures for determining silicon dioxide, calcium oxide, and magnesium oxide

Adopted as Standard:

Spec. for Masonry Cement (C 91 - 38 T), this recommendation being approved by unanimous consent with the following changes subject to favorable letter ballot of Committee C-1,[24] since the report as preprinted had recommended that the method as revised be published as tentative:

Section 6.—Change the last sentence from the form as preprinted to read as follows by the addition of the italicized words and the omission of the figure in brackets:

Nonstaining cement shall contain not more than 0.03 per cent of soluble "alkali" when tested in accordance with Section [27] *22 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114) of the American Society for Testing Materials.*

Section 27.—Omit the staining test included in the revision as preprinted.

Chemical Analysis of Portland Cement (C 114 - 39 T), adoption of Sections 1 to 9 with the following revision in Section 5, not preprinted, accepted by unanimous consent:

Section 5.—Change the first eight sentences from their present form to read as follows:

5. Weigh 1 to 3 g. of the sample (Note 1) and 10 g. of NH_4NO_3 into a 150-ml. beaker. Mix the contents, treat with 10 ml. of HNO_3 (sp. gr. 1.42), and stir quickly, using the flattened end of a glass rod to crush lumps of cement, until the cement is completely decomposed and the thick gel of SiO_2 is broken up. Cover the beaker with a watch glass, place it on a water bath or a hot plate at the approximately same temperature for 15 to 20 min., and stir the contents occasionally during the heating. Add 20 ml. of hot water to the beaker and stir the contents. If the cement contains an appreciable amount of manganese as shown by the presence of a red or brown residue, add a few milliliters of $NaNO_2$ solution (5 per cent) to dissolve this residue. Boil the contents of the beaker until all nitrous fumes are completely expelled. This should not take more than 5 min., and water should be added to replace any lost by evaporation. Filter into a 500-ml. Erlenmeyer flask under suction and with a platinum cone to support the filter and wash the residue of SiO_2 until the volume of filtrate and washings is about 150 ml.

Revision of Standard Methods of Chemical Analysis of Portland Cement (C 114 - 39) by the adoption of a new alternate method for the determination of phosphorus pentoxide and a new method for the determination of chloroform-soluble organic substances, not preprinted, accepted by unanimous consent subject to favorable letter ballot of Committee C-1.[25]

Revision of Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 - 39), not preprinted, accepted by unanimous consent subject to favorable letter ballot of Committee C-1.[26]

Section 2.—Change Paragraph (*a*) from its present form to read as follows by the addition of the italicized words:

(*a*) Samples for purpose of tests shall weigh at least 4 lb. *each when they are to be composited; single test samples, on which all specified tests are to be made, shall weigh at least 8 lb.*

Sections 2 (b), 3, 4 (a) and (c).—Change the number of barrels of cement represented by the test sample from "300 bbl." to read "400 bbl."

Section 4.—Combine with Section 3, relettering Paragraph (*b*) as (*c*) and change the second sentence of present Paragraph (*b*) from its present form: namely,

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[24] Committee C-1 reported results of the letter ballot vote as follows: Of a total voting membership of 89, 73 members returned their ballots, of whom 67 voted affirmatively, 0 negatively.

[25] Committee C-1 reported results of the letter ballot vote as follows: Of a total voting membership of 89, 73 members returned their ballots, of whom 67 voted affirmatively, 0 negatively.

[26] Committee C-1 reported results of the letter ballot vote as follows: Of a total voting membership of 89, 73 members returned their ballots, of whom 66 voted affirmatively, 2 negatively.

Tubes inserted horizontally may be used where the construction of the storage permits.

to read as follows:

This method may be used only when other methods cannot be applied, and great care must be exercised in order to obtain samples that completely represent the contents of the bin.

Section 5.—Designate this section as Paragraph 4 (*a*) and add a new Paragraph (*b*) to read as follows:

(*b*) Composite samples for the tests as required in Section 6 shall be prepared by arranging all test samples in groups, each group representing the number of barrels required by the test or tests for which the composite is intended. From each of the test samples in a group, equal portions shall be taken, sufficient in amount to form a composite large enough to permit the making of the required physical or chemical determination. The composite sample thus prepared shall be thoroughly mixed before using.

New Section.—Add a new Section 5 to read as follows:

5. *Number of Physical and Chemical Tests.*—(*a*) All required physical tests shall be made on each test sample, taken as specified in Section 3, from trucks and cars, no test sample to represent more than one car.

(*b*) Physical tests on samples taken from bins, boats, warehouses, etc., shall be required as follows:

Time of setting	each test sample representing each 400 bbl.
Fineness	composite sample representing each 800 bbl.
Strength	composite sample representing each 800 bbl.
Autoclave expansion	composite sample representing each 2000 bbl.

(*c*) Chemical determinations shall be required as follows:

SO_3	composite sample representing each 800 bbl.
All specified chemical determinations (except SO_3)	composite sample representing each 4000 bbl.

(*d*) When the total number of barrels sampled is less than that specified for any one of the above test or composite samples, all of the respective physical and chemical tests shall be made on the quantity sampled.

Section 10.—Change the first sentence to read as follows by the addition of the italicized word:

The quantities of dry materials to be mixed at one time shall be 500 g. for neat cement mixtures, and not less than 1000 nor more than 1200 g. for *briquet* mortar mixtures.

Figure 3.—Change the caption for Fig. 3 (*a*) to read as follows by the addition of the italicized words:

(*a*) Shrinkage Cracks, *Shown Above,* Due to Exposure of Pats to Dry Air During Setting *Are not to Be Considered as Evidences of Unsoundness.*

Papers:

The following papers were presented:

Some Properties of Mortar in Masonry—F. O. Anderegg, presented from manuscript by the author.

Tests of 106 Commercial Cements for Sulfate Resistance—Dalton G. Miller and Philip W. Manson, presented from manuscript by Mr. Miller.

A Method of Measuring Thermal Diffusivity and Conductivity of Stone and Concrete—W. T. Thomson, presented from manuscript by the author.

The Theory of Concrete Creep—William R. Lorman, presented, in the absence of the author, by H. S. Meissner.

Autogenous Volume Changes of Concrete—Harmer E. Davis, presented, in the absence of the author, by M. A. Swayze.

THIRTEENTH SESSION—FATIGUE, CORROSION-FATIGUE

Thursday, June 27, 9.30 a.m.

Session Co-Chairmen: President H. H. Morgan; R. E. Peterson.

Research Committee on Fatigue of Metals:

Progress report presented from manuscript by H. F. Moore, chairman.

Papers:

The following papers were presented:

Fatigue of Porous Metals—Claus G. Goetzel and Richard P. Seelig, presented by Mr. Seelig.

Fatigue Strength of 2-in. Diameter Axles with Surfaces Metal Coated and Flame Hardened—O. J. Horger and T. V. Buckwalter, presented by Mr. Horger.

Fatigue Tests on Zinc-Coated Steel Wire—D. G. Watt, presented from manuscript by the author.

A Pulsating Tension-Fatigue Machine for

Small Diameter Wire—John N. Kenyon, presented by the author.

The Effect of Protective Coatings on the Corrosion-Fatigue Strength of Steel—Thomas J. Dolan and Hugh H. Benninger, presented, in the absence of the authors, by H. F. Moore.

A Corrosion-Fatigue Test to Determine the Protective Qualities of Metallic Platings—John N. Kenyon, presented by the author.

FOURTEENTH AND FIFTEENTH SESSIONS —SYMPOSIUM ON SPECTROGRAPHIC ANALYSIS

Thursday, June 27, 9.30 a.m. and 2 p.m., respectively

Session Co-Chairmen: Vice-President G. E. F. Lundell; H. V. Churchill.

The Symposium on Spectrographic Analysis represented a frank interchange of views between the engineer or metallurgist who develops and uses metallurgical specifications on the one hand, and the analytical spectrographer who, on the other, checks such metals and alloys for conformance to compositional requirements. The subject was introduced by a paper by Thomas A. Wright on "Variables *versus* Constants in Emission Spectrography." Discussion then took place under two headings as follows:

Morning Session.—Accuracy Requirements *versus* Reproducibility Possibilities, participated in by:

W. H. Bassett, Jr.
E. B. Ashcraft
H. H. Stout, Jr.
Harry A. Bright
C. B. Frances
M. L. Fuller
G. J. Steele
A. E. Ruehle
A. W. Petrey
O. S. Duffendack
Lester W. Strock

Afternoon Session.—Interpretation of Results, participated in by:

C. C. Nitchie
A. C. Downes
Morris Slavin
A. W. Kenny
W. R. Koch
A. J. Phillips
T. S. Fuller
J. R. Townsend
Sam Tour
W. H. Finkeldey
E. E. Thum

In addition there was a considerable amount of spontaneous discussion, in which quite a number of those in attendance participated.

The symposium was supplemented by a separate session on Spectrochemical and Microanalytical Procedures held on Wednesday afternoon at 2 o'clock, under the chairmanship of B. F. Scribner, at which time the following illustrated talks were given:

Spectrochemistry of Iron and Steel—H. B. Vincent; illustrated by motion pictures.

Applications of Spectroscopy, Infrared and X-rays in Chemical Analysis—J. D. Hanawalt; illustrated by colored slides.

Industrial Applications of Microchemistry—G. L. Royer; illustrated by motion pictures.

SIXTEENTH SESSION—EFFECT OF TEMPERATURE, CREEP

Thursday, June 27, 2 p.m.

Session Chairman: H. J. French.

Joint Research Committee on Effect of Temperature on the Properties of Metals:

Report presented by N. L. Mochel, chairman, and the following papers, appended to the report, were presented as indicated:

Study of Effects of Variables on the Creep Resistance of Steels—H. C. Cross and J. G. Lowther, presented by Mr. Cross.

List of Published Reports with Reference to Tests on 0.35 per cent Carbon Steel K20—prepared by P. E. McKinney, A. B. Bagsar, E. L. Robinson and J. S. Worth, presented by title.

Papers:

The following papers were presented:

High-Speed Tension Tests at Elevated Temperatures—M. Manjoine and A. Nadai, presented by Mr. Manjoine.

The Creep Strength of 17 Low-Alloy Steels at 1000 F.—R. F. Miller, W. G. Benz and W. E. Unverzagt, presented by Mr. Miller.

Some Effects of Composition and Heat Treatment on the High Temperature Rupture Properties of Ferrous Alloys—R. H. Thielemann, presented by the author.

High Temperature Rupture and Creep Tests —Ernest L. Robinson, presented by the author.

SEVENTEENTH SESSION—PETROLEUM PRODUCTS, BITUMINOUS MATERIALS, ROAD MATERIALS, SOILS

Thursday, June 27, 8 p.m.

Session Co-Chairmen: H. S. Mattimore; E. F. Kelley.

Committee D-2 on Petroleum Products and Lubricants:

Report presented, in the absence of the chairman, by R. P. Anderson, and the following actions taken:

Accepted as Tentative:

Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) with the following changes not preprinted, approved by unanimous consent, subject to favorable letter ballot of Committee D-2.[27]

Section 2 (a).—Change the second sentence from its form as preprinted to read as follows by the addition of the italicized word and figures:

The tube shall be 140 ± 1 mm. in length and 14 ± 1 mm. in *outside* diameter, and shall be calibrated at the 5 ± *0.2*-ml. and 10 ± *0.2*-ml. liquid levels.

Test for Dropping Point of Lubricating Grease

Method of Calculating Viscosity Index

Revisions of:

Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 39 T), with the following additional change, approved by unanimous consent, subject to favorable letter ballot of Committee D-2:[28]

Section 2 (c).—Change the first sentence to read as follows by the addition of the italicized word:

A molten or solid metal bath with suitable arrangements for heating to a *uniform* temperature of 1022 F.

Test for Distillation of Plant Spray Oils (D 447 - 39 T)

Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 39 T)

Test for Gum Stability of Gasoline (D 525 - 39 T), with the following changes not preprinted approved by unanimous consent, subject to favorable letter ballot of Committee D-2:[29]

Figure 1.—Change the description, "Octagon $2\frac{3}{4}$" Across Flats" to read "Octagon $2\frac{3}{8}$" Across Flats."

Section 2 (a).—Change the second sentence from its present form: namely,

The composition of the gasket shall be approximately 80 per cent lead, 10 per cent antimony, and 10 per cent tin.

to read as follows:

Any suitable gasket may be used that will not react with oxygen.[a]

Spec. for Gasoline (D 439 - 37 T), with the following changes not preprinted approved by unanimous consent, subject to favorable letter ballot of Committee D-2.[30]

Table I.—Change the heading over the distillation temperatures to read as follows:

Minimum Percentages to be Evaporated at
Temperatures, deg. Fahr., Shown Below
10 per cent 50 per cent 90 per cent

Change octane numbers of 75, 67, and 45 to read 77, 70, and 50, respectively, wherever they occur in the table.

Change the reference to "1937" to read "1940" in footnotes "*b*" and "*d*".

Test for Distillation of Crude Petroleum (D 285 - 36), with the following change not preprinted approved by unanimous consent, subject to favorable letter ballot of Committee D-2:[31]

Special Procedure.—Introduce a new Section 8 to read as follows:

8. *Special Procedure for Obtaining Large Volumes of Distillate.*—If it is necessary to obtain quantities of distillate large enough for octane ratings or other special tests, the

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[a] Reactivity with oxygen is determined by the following test: Place the gasket under test in the bomb in the absence of gasoline and use a gasket of similar composition in the attachment of the cover. Fill the bomb with oxygen to a pressure of 100 psi. and immerse the bomb in a bath maintained at a temperature of 211.5 ± 0.5 F. If the pressure does not drop more than 2 lb. in a 24-hr. period, the gasket may be considered nonreactive in the presence of oxygen.

[27] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 94, 62 members returned their ballots, of whom 41 voted affirmatively, 0 negatively.

[28] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 94, 62 members returned their ballots, of whom 61 voted affirmatively, 0 negatively.

[29] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 94, 62 members returned their ballots, of whom 51 voted affirmatively, 0 negatively.

[30] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 94, 62 members returned their ballots, of whom 53 voted affirmatively, 0 negatively.

[31] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 94, 62 members returned their ballots, of whom 52 voted affirmatively, 1 negatively.

following procedure may be used: A charge of crude oil shall be distilled in equipment of suitable size to provide the desired amount of naphtha distillate in a single run or in a small number of runs. The equipment shall be so designed and operated that it duplicates both the yield and the quality of the distillate obtained by use of the regular procedure with a 300-ml. charge. The following criteria shall be considered in deciding whether the two procedures give equivalent results:

(*a*) Yields of naphtha distillate shall not differ by more than 1 per cent.

(*b*) The distillation curves of the naphtha fractions shall not differ at any point by more than 2.0 per cent.

Adopted as Standard:

Test for Unsulfonated Residue of Plant Spray Oils (D 483 - 38 T), as revised

Spec. for Stoddard Solvent (D 484 - 38 T), as revised

Revisions of:

- Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 38)
- Test for Distillation of Natural Gasoline (D 216 - 39)
- Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 36)
- Analysis of Grease (D 128 - 37)
- Test for Knock Characteristics of Motor Fuels (D 357 - 39), with the following additional change not preprinted approved by unanimous consent, subject to favorable letter ballot of Committee D-2:[32]

Section 3 (*t*).—Change the last sentence to read as follows by the addition of the italicized words and the omission of the words and figures in brackets:

The use of a short straight-through muffler, *or equivalent* with passage [1¼ in. in diameter] *not less than the diameter of the exhaust pipe* is permissible.

Appendix II.—Change the heading of the first column of Table I from its form as preprinted, "Approximate Altitude, ft." to read "Nearest Altitude, ft."

Test for Precipitation Number of Lubricating Oils (D 91 - 35) and Test for Water in Petroleum Products by Means of Centrifuge (D 96 - 35), with the following change not preprinted approved by unanimous consent, subject to favorable letter ballot of Committee D-2:[33]

Centrifuge Tubes.—Change the calibration requirements in the range 1 to 3 ml. to read as follows:

Range, ml.	Scale Division, ml.	Limit of Error, ml.	Numbered, ml.
1 to 2..........	0.1	0.1	2
2 to 3..........	0.2	0.1	3

Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 30), with the following change not preprinted approved by unanimous consent, presented jointly with Committee D-4 on Road Materials, subject to favorable letter ballot of Committee D-2:[34]

Section 5. (*a*).—In the note following this section change the last sentence to read as follows, by the addition of the italicized words and figures and the omission of those in brackets:

This tube shall be graduated from 0 to [25 ml.] *2 ml.* in 0.1-ml. divisions *and from 2 to 25 ml. in 0.2-ml. divisions* (see Fig. 3 (*c*) of the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials).

Publication as Information Only:

Proposed Method of Test for Aniline Point of Petroleum Products

Proposed Method of Test for Ignition Quality of Diesel Fuels

Diesel-Fuel-Oil Classification

The revised Method of Test for Neutralization Number of Petroleum Products and Lubricants was recommended and accepted for publication as information rather than as a revision of Tentative Method D 188 - 27 T, as indicated in the preprinted report, subject to favorable letter ballot of Committee D-2.[35]

Sectional Committee Z11 on Petroleum Products and Lubricants:

Report presented from manuscript, in the absence of the chairman, by T. G. Delbridge, vice-chairman.

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[32] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 94, 62 members returned their ballots, of whom 48 voted affirmatively, 0 negatively.

[33] Committee D-2 reported the following results of the letter ballot vote of a total of 62 ballots returned from a voting membership of 94: D 91, affirmative 54, negative 1; D 96, affirmative 57, negative 1.

[34] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 94, 62 members returned their ballots, of whom 58 voted affirmatively, 0 negatively.

[35] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 94, 62 members returned their ballots, of whom 56 voted affirmatively, 2 negatively.

Committee D-4 on Road and Paving Materials:

Report presented by E. F. Kelley, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Materials for Stabilized Base Course
Spec. for Materials for Stabilized Surface Course

Adopted as Standard:

Spec. for Quick-Setting Emulsified Asphalt (for Penetration and Surface Treatment) (D 401 - 36 T)
Spec. for Asphalt Plank (D 517 - 38 T)
Revision of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 30), this change, as given above,[36] being presented jointly with Committee D-2 on Petroleum Products and Lubricants, was accepted by unanimous consent.

Revision of Tests for Emulsified Asphalts (D 244 - 39), with the change given below, as recommended jointly with Committee D-8 on Bituminous Waterproofing and Roofing Materials, was accepted by unanimous consent.

Withdrawal of Tentative Standard:

Test for Abrasion of Gravel by Use of the Deval Machine (D 289 - 37 T).

Publication as Information Only:

Proposed Specifications for Preformed Expansion Joint Filler for Concrete
Proposed Methods of Testing Preformed Expansion Joint Fillers for Concrete

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented by J. M. Weiss, chairman, and the following actions taken:

Accepted as Tentative:

Revisions of Spec. for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 - 39 T).

Adopted as Standard:

Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (D 451 - 37 T)
Test for Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (D 452 - 37 T).
Methods of Testing Emulsified Asphalts (D 244 - 39), with the following change, not preprinted, accepted by unanimous consent, presented jointly with Committee D-4 on Road and Paving Materials.

Section 8 (f).—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

The trap shall be made of well-annealed glass constructed in accordance with Fig. 3 (*c*) and shall be graduated from 0 to [25 ml.] *2 ml.* in 0.1-ml. divisions, *and from 2 to 25 ml. in 0.2-ml. divisions.*

Committee D-18 on Soils for Engineering Purposes:

Report presented by H. F. Clemmer, chairman, and the following actions taken:

Accepted as Tentative:

Test for Moisture-Density Relations of Soil-Cement Mixtures
Wetting-and-Drying Test of Compacted Soil-Cement Mixtures
Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures
Revision of Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 - 35 T)

The recommendation for acceptance as tentative of the Method of Test for Water Absorption and Resistance to Plastic Flow of Mixtures of Soil and Emulsified Asphalt was discussed and on motion was referred to Committee E-10 on Standards.

Colored moving pictures illustrating the procedures followed in the testing of soils, both in the laboratory and in the field, were shown through the courtesy of C. A. Hogentogler.

Papers:

The following papers were presented:

A Study of the Recovery of the Bitumen from Soft Asphalt Cements by the Bussow Method—Carl Bussow, presented by the author.

Rheological Interpretation of Asphalt Tests

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[36] See p. 21.

—R. N. Traxler, H. E. Schweyer, and J. W. Romberg, presented from manuscript by Mr. Traxler.

The Measurement of the Deformation Properties of Asphalts—T. F. Ford and K. G. Arabian, presented from manuscript by Mr. Ford.

The Mechanics of Granular and Granular-Plastic Materials, with Special Reference to Bituminous Road Materials and Subsoils—V. A. Endersby, presented from manuscript by the author.

EIGHTEENTH SESSION—CONCRETE (continued from Twelfth Session)

SANFORD E. THOMPSON AWARD

Thursday, June 27, 8 p.m.

Session Chairman: Stanton Walker.

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by F. H. Jackson, chairman, and the following actions taken:

Accepted as Tentative:

Test for Efficiency of Materials for Curing Concrete

Test for Volume Change of Cement Mortar and Concrete

Revision of Test for Compressive Strength of Concrete (C 39 - 39)

Adopted as Standard:

Revision of Spec. for Concrete Aggregates (C 33 - 39), with the following change not preprinted approved by unanimous consent, subject to favorable letter ballot of Committee C-9:[37]

Section 11 (*k*).—Omit this section which reads as follows, as the method referred to has been discontinued:

(*k*) *Soft Fragments.*—Method of Test for Quantity of Soft Pebbles in Gravel, Standard Method T-8 of The American Association of State Highway Officials.

The chairman then introduced Sanford E. Thompson in whose honor the Sanford E. Thompson award had been established by Committee C-9 in honor of the first chairman of the committee, and for the purpose of stimulating research and to encourage investigations that extend the knowledge of concrete and concrete aggregates.

Mr. Thompson recalled briefly some of the early research activities of the committee and stressed the important progress made in its standardization work, particularly the development of testing procedures. He pointed out that the subject of the winning paper was directly in line with the original aims of Committee C-9, namely, the attainment of durable concrete. He stated that the authors had studied the effects of different constituents with volume changes and elasticities with special reference to durability in weathering and had incorporated basic factors considered by Committee C-9 to be essential in a paper meriting the award, namely, originality of approach, skill in execution, and stimulation to further work in an important field of concrete research.

Mr. Thompson then introduced the recipients of the award, Messrs. T. F. Willis and M. E. De Reus for their paper on "Thermal Volume Change and Elasticity of Aggregates and Their Effect on Concrete," presented at the 1939 annual meeting. On behalf of the committee Mr. Jackson then presented to the authors the certificate of the Sanford E. Thompson Award.

On behalf of the recipients Mr. DeReus expressed appreciation of the honor of the award.

Committee C-13 on Concrete Pipe:

Informal progress report presented, in the absence of the chairman, by E. F. Kelley. The report summarized the tentative revision of the Standard Specifications for Concrete Sewer Pipe (C 14-35), for Reinforced-Concrete Sewer

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[37] Committee C-9 reported results of the letter ballot vote as follows: Of a total voting membership of 68, 55 members returned their ballots, of whom 52 voted affirmatively, 1 negatively.

Pipe (C 75 - 35) and for Reinforced-Concrete Culvert Pipe (C 76 - 37) to be submitted to Committee E-10 on Standards subsequent to the Annual Meeting.

Papers:

The following papers were presented:

The Soundness of Chert as Measured by Apparent Specific Gravity and Absorption—Charles E. Wuerpel and E. P. Rexford, presented by Mr. Wuerpel.

The Influence of Test Methods on Moisture Absorption and Resistance of Coarse Aggregate to Freezing and Thawing—F. N. Wray and H. J. Lichtefeld, presented, in the absence of the authors, by T. F. Willis.

Designing Concrete Mixtures for Pavements—W. F. Kellermann, presented from manuscript, in the absence of the author, by F. H. Jackson.

Measuring Changes in Physical Properties of Concrete by the Dynamic Method—W. T. Thomson, presented by the author.

NINETEENTH SESSION—TEXTILES, SOAPS, PAINT, TIMBER, PAPER, FUELS

Friday, June 28, 9.30 a.m.

Session Chairman: H. J. Ball.

Committee D-13 on Textile Materials:

Report presented by W. H. Whitcomb, secretary, and the following actions taken:

Accepted as Tentative:

- Spec. for All Wool, All Cotton, and Wool and Cotton Blanketing (Household)
- Testing Woven Asbestos Cloth
- Testing and Tolerances for Glass Yarn
- Testing and Tolerances for Woven Glass Tapes
- Testing and Tolerances for Woven Glass Fabrics
- Testing and Tolerances for Woven Glass Tubular Sleeving and Braids
- Test for Resistance of Fabrics to Water, subject to review and final decision by the Executive Committee since the apparatus for the water penetration (drip) method is similar to that covered by a U. S. patent.[38]
- Test for Resistance of Fabrics and Yarns to Moths
- Test for Shrinkage of Grease Wool (Laboratory Scale Operations)
- Revisions of:
  - Spec. for Bleached Wide Cotton Sheeting (D 503 - 38 T)
  - Spec. and Test for Fineness of Wool Tops (D 472 - 37 T)
  - Test for Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide (D 539 - 39 T)
  - Definitions of Terms Relating to Textile Materials (D 123 - 39 T)
  - Spec. for Textile Testing Machines (D 76 - 39)
  - Spec. and Test for Asbestos Roving for Electrical Purposes (D 375 - 37)
  - Spec. and Test for Asbestos Yarns (D 299 - 37)
  - Spec. and Test for Asbestos Tape for Electrical Purposes (D 315 - 37)
  - Tests and Tolerances for Cotton Sewing Threads (D 204 - 39)
  - Tests and Tolerances for Tire Cord, Woven and on Cones (D 179 - 38)

Adopted as Standard:

- Spec. for Terry (Turkish) Toweling (D 505 - 38 T)
- Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 39 T)
- Test for Fiber Length of Wool (D 519 - 38 T)
- Testing Wool Felt (D 461 - 38 T), as revised
- Definitions of Terms Relating to Textile Materials (D 123 - 39 T)
- Revisions of:
  - Testing and Tolerances for Cotton Yarns and Thread (D 180 - 37)
  - Testing and Tolerances for Hose Ducks and Belt Ducks (D 181 - 36)
  - Testing Pile Floor Covering (D 418 - 39)
  - Spec. for Textile Testing Machines (D 76 - 39)
  - Spec. and Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 - 34)

Withdrawal of Tentative Revision of Standard:

- Testing Woven Textile Fabrics (D 39 - 39)

Committee D-12 on Soaps and Other Detergents:

Report presented by H. P. Trevithick, chairman, and the following actions taken:

Accepted as Tentative:

- Spec. for Olive Oil Solid Soap
- Spec. for Salt-Water Soap

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[38] **The Executive Committee approved the acceptance of these methods for publication as tentative.**

Spec. for Tetrasodium Pyrophosphate (Anhydrous)
Spec. for Sodium Sesquisilicate

Revisions of:
  Spec. for Palm Oil Bar Soap (D 535 - 39 T)
  Sampling and Chemical Analysis of Special Detergents (D 501 - 39 T)
  [Definitions of Terms Relating to Soaps and Other Detergents (D 459 - 39 T)
  Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 39)

Reversion of Standard to Tentative:

Definitions of Terms Relating to Soaps and Other Detergents (D 459 - 39), as revised

Committee D-17 on Naval Stores:

Report presented, in the absence of the chairman, by V. E. Grotlisch.

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by H. E. Smith, chairman. The recommendations on standards appearing in the report were presented by M. Rea Paul, secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Titanated Lithopone, to be added, when adopted, to Standard Spec. D 477 - 39

Spec. for Ultramarine Blue, to replace Standard Spec. D 262 - 28

Spec. for Carbon Black

Test for Consistency of Enamel Type Paints, with two editorial changes in Section 2, changing "shearing stress" to read "weight," and "rate of shear" to read "speed."

Test for Phthalic Anhydride Content of Alkyd Resin Solutions

Testing Liquid Driers

Revision of Definitions of Terms Relating to Paint (D 16 - 24), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee D-1,[39] this recommendation resulting from a change by the committee in the report as preprinted which had recommended the immediate adoption as standard of the revised definitions:

Amorphous and Crystalline.—Delete the definitions of these two terms.

Hue.—Change from the form as preprinted to read as follows:

Hue.—An attribute of color which determines whether the color is red, yellow, green, blue, purple, or an intermediate.

Paint.—Change the first paragraph of this definition from the form as preprinted to read as follows:

Paint.—A pigmented liquid composition which is converted to an opaque solid filled after application as a thin layer.

Shade.—Change from the form as preprinted to read as follows:

Shade.—A term descriptive of a lightness difference between surface colors, the other attributes of color being essentially constant. A lighter shade of a color is one that has higher lightness but approximately the same hue and saturation; and a darker shade is one that has a lower lightness.

NOTE.—Primarily the term "shade" is derived from shadow and designates a change in appearance analogous to that produced by a local reduction in illumination. It should, therefore, when strictly used, express only the change toward a darker color. Shade of a color has been defined by several authorities as the mixture of black with that color, thus establishing its opposite character to "tint," but by extension of its relative sense it has been frequently and widely used to include lighter shades by use of the adjective "lighter" or "paler." Although such expressions apparently involve a contradiction, it is clear that while we may have a shade of a color, or darker color of the same sort, it is easy to conceive of another shade not quite so dark and therefore lighter.

Tone and Water.—Delete the definitions of these two terms.

Varnish.—Change from the form as preprinted to read as follows:

Varnish.—A liquid composition which is converted to a transparent or transluscent solid filled after application as a thin layer.

Revision of Chemical Analysis of White Linseed Oil Paints (D 215 - 37)

Revision of Methods of Sampling and Testing Turpentine (D 233 - 36), with the following change in the revision as preprinted to clarify the section and avoid any possible misunderstanding:

Section 9 (d).—Change from the form as preprinted to read as follows:

(*d*) *Thermometer.*—An A.S.T.M. turpentine distillation thermometer, 3-in. partial immersion, having a range of 148 to 182 C., conforming to the requirements for this thermometer as prescribed in the Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1 - 40 T) shall be used.[a]

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[39] **Committee D-1 reported results of the letter ballot vote as follows: Of a total voting membership of 185, 124 members returned their ballots, of whom 114 voted affirmatively, 1 negatively.**

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[a] The short total immersion turpentine distillation thermometer, having a range of 145 to 200 C., conforming to the requirements for this thermometer as previously prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1 - 39) of the American Society for Testing Materials, may be used in lieu of the new standard 3-in. partial immersion turpentine distillation thermometer, if desired.

Adopted as Standard:

Spec. for Zinc Dust (Metallic Zinc Powder) (D 520 - 39 T)
Chemical Analysis of Zinc Dust (Metallic Zinc Powder) (D 521 - 39 T), as revised
Test for Reactivity of Paint Liquids (D 479 - 38 T)

Revisions of:

Spec. for Leaded Zinc Oxide (D 80 - 24)
Spec. for Mineral Iron Oxide (D 84 - 27)
Spec. for Ocher (D 85 - 27)
Spec. for Chrome Yellow (D 211 - 27)
Spec. for Pure Chrome Green (D 212 - 27)
Spec. for Reduced Chrome Green (D 213 - 27)
Spec. for Prussian Blue (D 261 - 39)
Spec. for Chrome Oxide Green (D 263 - 28)
Spec. for Reduced Para Red (D 264 - 39)
Spec. for C. P. Para Red Toner (D 475 - 39)
Spec. for C. P. Zinc Yellow (Zinc Chromate) (D 478 - 39)
Sampling and Analysis of Shellac (D 29 - 39)
Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 - 37)
Spec. for Normal Butyl Acetate (88 to 92 per cent Grade) (D 303 - 33)
Spec. for Butanol (Normal Butyl Alcohol) (D 304 - 33)
Spec. for Amyl Alcohol (Synthetic) (D 319 - 33)
Spec. for Butyl Propionate (90 to 93 per cent Grade) (D 320 - 33)
Spec. for Ethyl Lactate (Synthetic) (D 321 - 33)

Withdrawal of Standards:

Spec. for Ultramarine Blue (D 262 - 28)
Definitions of Terms Relating to Paint (D 16 - 24), this recommendation, not preprinted, accepted by unanimous consent subject to favorable letter ballot of Committee D-1.[40]

A report on "Accelerated Weathering Tests of Enamels on Steel and Correlation with Outdoor Exposure," prepared by Group 2 on Oleoresinous Type Enamels, of Subcommittee VII, and submitted by Committee D-1 for inclusion as an appendix to its report was accepted, subject to review by Committee E-6 on Papers and Publications.

Committee D-7 on Timber:

Report presented, in the absence of the chairman, by the chair and the editorial change in the Tentative Method of Test for Tar Acids in Creosote and Creosote - Coal Tar Solutions (D 453 - 39 T) was accepted.

Committee D-6 on Paper and Paper Products:

Report presented by L. S. Reid, secretary, and the following actions taken:

Accepted as Tentative:

Sampling Paper and Paper Products
Test for Casein in Paper (Qualitative)
Test for Alpha-, Beta-, and Gamma-Cellulose in Paper
Test for Starch in Paper
Test for Paraffin Content of Waxed Paper
Test for Opacity of Paper and Paper Products
Test for Ash Content of Paper and Paper Products

Committee D-3 on Gaseous Fuels:

Report presented, in the absence of the chairman, by W. A. Selvig.

Committee D-5 on Coal and Coke:

Report presented, in the absence of the chairman, by H. C. Porter, vice-chairman, and the following actions taken:

Accepted as Tentative:

Revision of Methods of Sampling Coals Classed According to Ash Content (D 492 - 38 T)

Adopted as Standard:

Revision of Methods of Sampling Coal for Analysis (D 21 - 16)
Revision of Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 37)

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[40] Committee D-1 reported results of the letter ballot vote as follows: Of a total voting membership of 185, 124 members returned their ballots, of whom 111 voted affirmatively, 0 negatively.

In presenting the report, Mr. Porter announced that Committee D-5 at its meeting on June 27 had taken action to modify the report as preprinted, this action requiring the following changes in the report as preprinted:

> Delete from page 1 of the report the second recommendation affecting standards proposing the adoption as standard of the Tentative Definition of the Term Coke (D 121 - 26 T), as revised; also eliminate this item from the letter ballot table on page 2.
>
> Under activities of Subcommittee II on Nomenclature and Definitions appearing on page 2 of the report as preprinted, substitute for the first sentence which reads as follows:
>
> This subcommittee recommends for adoption as standard a revised form of the Tentative Definition of the Term Coke (D 121 - 26 T) as mentioned earlier in this report.
>
> the following:
>
> At a meeting of Committee D-5 held in Detroit, Mich., on March 5, 1940, this subcommittee recommended for adoption as standard a revision of the Tentative Definition of the Term Coke (D 121 - 26 T), as follows:
>
> *Coke.*—(*a*) The solid, cellular, coherent residue from destructive distillation of bituminous coal, said residue having such physical structure as results from the hardening of a fused or semiliquid mass.
>
> (*b*) If used with appropriate qualifying adjective, a similar residue from petroleum, pitch, and some other carbonaceous materials.
>
> This recommendation was submitted to letter ballot of Committee D-5 which consists of 44 members; 37 members returned their ballots, of whom 32 have voted affirmatively, 2 negatively, and 3 members marked their ballots "not voting." However, in deference to a written request dated June 24, 1940, from the Executive Committee of the Society addressed to the chairman of Committee D-5, it was voted at a meeting of Committee D-5 held on June 27, 1940, not to present this revised definition at the annual meeting of the Society.

The above change in the report as recommended by the committee was discussed, and on motion the report as amended was accepted with one dissenting vote.

Papers:

The following papers were presented:

The Creep Phenomenon in Ropes and Cords—Carlton G. Lutts and David Himmelfarb, in the absence of the authors, presented by title.

An Extraction Method for the Determination of Metals in Boiled Linseed Oil and Driers—Frank Gottsch and Benjamin Grodman, presented by Mr. Gottsch.

TWENTIETH SESSION—NON-FERROUS METALS

Friday, June 28, 9.30 a.m.

Session Chairman: T. S. Fuller.

Committee B-5 on Copper and Copper Alloys, Cast and Wrought

Report presented by C. H. Greenall, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Cartridge Brass Cartridge Case Cups

Spec. for Gilding Metal Sheet and Strip

Spec. for Gilding Metal Bullet Jacket Cups, with the following additional changes not preprinted accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[41]

> *Section 1.*—Change from the form as preprinted to read as follows:
>
> 1. These specifications cover one grade of gilding metal cups of the following types and classes for the manufacture of bullet jackets.
>
> *Type I.*—Caliber 0.30
> *Type II.*—Caliber 0.45
> *Type III.*—Caliber 0.50
> *Type IV.*—As specified in the contract or order
>
> *Class I.*—Not annealed
> *Class II.*—Annealed
> *Class III.*—As specified in the contract or order
>
> *Section 7.*—Change from the form as preprinted to read as follows:
>
> 7. (*a*) The grain size as determined on

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[41] Committee B-5 reported results of the letter ballot vote as follows: Of a total voting membership of 80, 54 members returned their ballots, of whom 30 voted affirmatively, 0 negatively.

the cups shall be as shown in the following table:

Class	Grain Size, mm. On Base[a]	Grain Size, mm. On Sidewall[b]
I	0.015 to 0.040	
II	0.015 to 0.050	0.015 to 0.040
III	As specified in the contract	

[a] On approximately the center of the base.
[b] Approximately midway of the length of the side wall.

(*b*) When viewed at a magnification of 75 × the average grain size of selected areas 79.8 mm. in diameter shall not exceed these limits.

Section 12.—Add the words "and class" after the word "type."

Spec. for Copper Rods and Bars, with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5.[42]

Section 1.—Add the following Paragraph (*c*) to this section:

(*c*) Type A material is processed to conform to the Rockwell hardness requirements specified in Section 6. Type B material is processed to conform to the tensile requirements prescribed in Table I.

Section 6.—Include headings of "minimum" and "maximum" in the table of Rockwell hardness numbers for the B and F Scales.

Section 9.—Change the electrical resistivity equivalents from the form as preprinted to read as follows:

Condition	Electrical Resistivity ohm (meter, gram)
Soft	0.15436
Hard	0.15775

Also change the electrical resistivity and conductivity equivalents in the note to read as follows:

Electrical Resistivity, ohm (meter, gram)	Conductivity, per cent
0.15436	99.30
0.15775	97.16

Spec. for Brass Wire

Spec. for Leaded High-Strength Yellow Brass (Manganese Bronze) Castings

Spec. for Miscellaneous Brass Tubes, with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[43]

Section 1.—Add the words "in straight lengths" at the end of the first sentence.

Table I.—For the designations of drawn temper change "quarter hard" to read "light drawn."

Section 5.—Change the second sentence from the form as preprinted to read as follows:

Light-drawn tubes shall be interpreted to mean tubes cold drawn to a reduction of area of 10 to 25 per cent. Hard-drawn tubes shall be interpreted to mean tubes cold drawn to a reduction of area of more than 25 per cent.

Table II.—Change the approximate Rockwell hardness values of annealed tubes for alloys Nos. 2, 3, and 4 to read as follows:

	Rockwell Hardness F-Scale Min.	Rockwell Hardness F-Scale Max.	Rockwell Hardness 30T Superficial Min.	Rockwell Hardness 30T Superficial Max.
Soft anneal	60	75	18	39
Light anneal	67	89	28	53

Section 10.—In the mercurous nitrate test add the phrase "when specifically required" at the beginning of the first sentence.

Section 13.—Omit the first sentence which reads: "No lot of tubes shall exceed the theoretical weight by more than 5.0 per cent.

Table IV.—Delete the tolerances for tubing $\frac{1}{8}$ in. and under in coils.

Revisions of:

Spec. for Brass Sheet and Strip (B 36 - 39 T)

Spec. for Copper-Silicon Alloy Plate and Sheet (B 96 - 39 T)

Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 39 T)

Spec. for Brass Pipe, Standard Sizes (B 43 - 39 T)

Classification of Cast Copper-Base Alloys (B 119 - 39 T)

Spec. for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 - 38 T)

Spec. for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 - 39 T)

Adopted as Standard:

Spec. for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 - 38 T)

Spec. for Phosphor Bronze Sheet and Strip (B 103 - 39 T)

Revisions of:

Spec. for Seamless Copper Boiler Tubes (B 13 - 33)

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[42] Committee B-5 reported results of the letter ballot vote as follows: Of a total voting membership of 80, 54 members returned their ballots, of whom 34 voted affirmatively, 0 negatively.

[43] Committee B-5 reported results of the letter ballot vote as follows: Of a total voting membership of 80, 54 members returned their ballots, of whom 37 voted afirmatively, 0 negatively.

Spec. for Sheet Copper-Silicon Alloy (B 97 - 39)
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 39)
Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 - 39)
Spec. for Steam or Valve Bronze Castings (B 61 - 36)

Reversion of Standards to Tentative:

Spec. for Cartridge Brass (B 19 - 29), as revised
Spec. for Naval Brass Rods for Structural Purposes (B 21 - 29), as revised, with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[44]

Table II.—Designate the present requirements for minimum elongation in 2 in. as applying to material of Grades A and B and add the following requirements for Grade C:

Diameter or Thickness, in.	Grade C Elongation in 2 in., min., per cent
Rods and Bars, Soft:	
1 and under	20
1.001 to 2.50	25
2.501 and over	30
Rods and Bars, Half Hard:	
0.5 and under	12
0.501 to 1	15
1.001 to 2.50	20
2.501 to 3.50	25
3.501 and over	25
Rods and Bars, Hard:	
1 and under	10
1.001 to 2.50	15
2.501 to 3.50	20
3.501 and over	20
Shapes, Soft, all Sizes	20

Spec. for Seamless Copper Tubes (B 75 - 30), as revised. It was announced that additional revisions in these specifications would be submitted to the Society through Committee E-10 on Standards subsequent to the annual meeting.

Withdrawal of Standards:

Spec. for Cartridge Brass Disks (B 20 - 29)
Spec. for Leaded-Nickel-Brass and Leaded-Nickel-Bronze (Nickel-Silver) Alloys in Ingot Form for Sand Castings (B 123 - 39 T)

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by E. E. Thum, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Pig Lead, to replace immediately Spec. B 29 - 35.

Adopted as Standard:

Spec. for Lead-Coated Copper Sheets (B 101 - 35 T)

Papers:

The following papers were presented:

Proportional Limit Tests on Copper Alloys—Cyril Stanley Smith, presented by the author.

Internal Hydraulic Bursting Tests of Lead Cable Sheathing—Howard S. Phelps, Albert M. Gates, and Frank Kahn, presented by Mr. Phelps.

Bursting Tests and Tension Tests for Lead Cable Sheathing—H. F. Moore, C. W. Dollins, and W. J. Craig, presented by Mr. Dollins.

The Testing of Lead Cable Coverings—W. H. Bassett, Jr. and C. J. Snyder, presented from manuscript by Mr. Snyder.

TWENTY-FIRST SESSION—NON-FERROUS METALS

Friday, June 28, 2 p.m.

Session Chairman: President H. H. Morgan.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by J. R. Townsend chairman, and the following action taken:

Accepted as Tentative:

Revision of Spec. for Magnesium-Base Alloy Die Castings (B 94 - 39 T)

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by Sam Tour, chairman, and the following actions taken:

Accepted as Tentative:

Test for Weight of Coating on Anodically Coated Aluminum

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[44] Committee B-5 reported results of the letter ballot vote as follows: Of a total voting membership of 80, 54 members returned their ballots, of whom 35 voted affirmatively, 0 negatively.

Test for Sealing of Anodically Coated Aluminum
Revisions of:
  Spec. for Magnesium-Base Alloy Ingot for Remelting (B 93 - 39 T)
  Spec. for Magnesium-Base Alloy Sand Castings (B 80 - 38 T)
  Spec. for Magnesium-Base Alloy Forgings (B 91 - 38 T)
  Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 38 T)
  Spec. for Magnesium-Base Alloy Sheet (B 90 - 38 T)
  Spec. for Aluminum Sheet and Plate (B 25 - 38 T)
  Spec. for Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese) (B 78 - 39 T)
  Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 - 38 T)
  Spec. for Aluminum-Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum - Copper - Magnesium - Manganese) (B 89 - 39 T)
  Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 - 39 T
  Spec. for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 - 39 T)

In the 10 specifications immediately above, the following revision, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee B-7:[45]

Methods of Testing.—Add to each of the 10 specifications the following as a new section on methods of testing:

The tension tests shall be made in accordance with the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.

Figure 1.—In these specifications change the note regarding the radius at the end of the reduced section from "radius not less than $\frac{1}{8}$ in." to read "radius not less than $\frac{3}{8}$ in."

Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors:

Report presented by J. H. Foote, chairman, and the following actions taken:

Accepted as Tentative:

Revision of Spec. for Soft Rectangular and Square Copper Wire for Electrical Conductors (B 48 - 39 T)

Adopted as Standard:

Spec. for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (B 116 - 39 T)

Revision of Spec. for Hard-Drawn Copper Wire (B 1 - 39) and Spec. for Medium-Hard-Drawn Copper Wire (B 2 - 39), comprising the following change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-1,[46] this change being recommended to bring the requirements on permissible variations in diameter into agreement with those prescribed in Spec. for Soft or Annealed Copper Wire (B 3 - 39):

Section 5 (b).—Change from its present form to read as follows:

(*b*) Within the range of diameters included in Table I the wire shall not vary from the specified diameter by more than plus or minus 1 per cent.

The proposed Tentative Specifications for Bare Rope-Lay-Stranded and Bunch-Stranded Soft Copper Cables for Electrical Conductors were withdrawn from the report by the committee as it was announced that these specifications were to be given further consideration, looking toward their publication as tentative through submission to Committee E-10 on Standards.

The proposed tentative revision of the Specifications for Hot-Rolled Copper Rods for Electrical Purposes (B 49 - 39) comprising a proposed procedure for the twist test, was withdrawn from the report by the committee.

Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys:

Report presented by Dean Harvey, chairman, and the following actions taken:

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[45] Committee B-7 reported the following results of the letter ballot vote of a total of 36 ballots returned from a voting membership of 44: B 25, B 78, B 79, and B 89, affirmative 30, negative 0; B 90, B 91, B 107, B 109, and B 126, affirmative 29, negative 0; B 80, affirmative 32, negative 0.

[46] Committee B-1 reported results of the letter ballot vote as follows: Of a total voting membership of 31, all members returned their ballots, of whom 28 voted affirmatively, 1 negatively.

Accepted as Tentative:

Testing Sleeves and Tubing for Radio Tube Cathodes

Adopted as Standard:

Test for Flexivity of Thermostat Metals (B 106 - 39 T), with the following additional changes not preprinted approved by unanimous consent, subject to favorable letter ballot of Committee B-4:[47]

Title.—Change from its present form to read: "Methods of Testing Thermostat Metals."

Section 1.—Add a new section on scope to read as follows:

1. These methods cover the procedures for testing thermostat metals.

Renumber present Sections 1 to 7 as Sections 2 to 8 and add a new heading "Flexivity" preceding new Section 2.

Section 4 (e).—Change from its form as preprinted to read as follows:

NOTE 1.—This corresponds to a permissible variation of plus or minus 0.66 per cent for specimens 0.015 in. in thickness, and plus or minus 0.2 per cent for specimens 0.050 in. in thickness.

New Sections.—Add new Sections 9 and 10 on "Electrical Resistivity" and "Change of Resistance with Temperature" to read as follows:

9. Electrical resistivity shall be determined in accordance with the Standard Method of Test for Resistivity of Metallic Materials (A.S.T.M. Designation: B 63) of the American Society for Testing Materials.

10. Change of resistance with temperature shall be determined in accordance with the Standard Method of Test for Change of Resistance with Temperature of Metallic Materials for Electrical Heating (A.S.T.M. Designation: B 70) of the American Society for Testing Materials.

A motion that the Tentative Method for Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (B 113 - 38 T) be approved for reference to letter ballot of the Society for adoption as standard failed to receive the necessary two-thirds vote of those voting at the session, the result of the vote being three affirmative and two negative.

Papers:

The following papers were presented:

Anodic Coatings Seen Through the Microscope—F. Keller, presented by the author.

Thickness of Anodic Coatings on Aluminum—Junius D. Edwards, presented by the author.

The Abrasion Resistance of Anodically Oxidized Coatings on Aluminum—H. G. Arlt, presented by the author.

Electrical Breakdown of Anodically Oxidized Coatings on Aluminum: A Means of Checking Thickness of Anodized Finishes—K. G. Compton and A. Mendizza, presented by Mr. Mendizza.

Creep of Aluminum Subjected to Bending at Normal Temperatures—Joseph Marin and L. E. Zwissler, presented by Mr. Marin.

TWENTY-SECOND SESSION—PLASTICS, ELECTRICAL INSULATING MATERIALS, RUBBER

Friday, June 28, 2 p.m.

Session Chairman: M. F. Skinker.

Committee D-20 on Plastics:

Report presented by W. E. Emley, chairman, and the following actions taken:

Accepted as Tentative:

Test for Flammability of Plastics
Test for Water Absorption of Plastics
Measuring Flow Temperatures of Thermoplastic Molding Materials, subject to review and final decision by the Executive Committee since the apparatus for the flow test is covered by a U. S. patent.[48]

Committee D-9 on Electrical Insulating Materials:

Report presented by T. Smith Taylor, chairman, and the following actions taken:

Accepted as Tentative:

Revisions of:
  Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 39 T)

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[47] Committee B-4 reported results of the letter ballot vote as follows: Of a total voting membership of 45, 34 members returned their ballots, of whom 29 voted affirmatively, 0 negatively.

[48] The Executive Committee approved the acceptance of this method for publication as tentative.

Tests for Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 39 T)
Spec. for Flexible Varnished Tubing Used in Electrical Insulation (D 372 - 37 T)
Spec. for Phenolic Laminated Sheet for Radio Applications (D 467 - 37 T)
Tests for Molded Materials Used for Electrical Insulation (D 48 - 39)
Tests for Electrical Insulating Oils (D 117 - 36)
Tests for Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39)
Tests for Laminated Tubes Used in Electrical Insulation (D 348 - 39)
Tests for Laminated Round Rods Used in Electrical Insulation (D 349 - 39)

Adopted as Standard:

Test for Conducting Paths in Electrical Slate (D 273 - 36 T)
Tests for Shellac Used for Electrical Insulation (D 411 - 37 T)
Revision of Tests for Electrical Insulating Oils (D 117 - 36)

Sectional Committee C59 on Electrical Insulating Materials:

Report presented by H. L. Curtis, chairman.

Committee D-11 on Rubber Products:

Report presented by O. M. Hayden, chairman, and the following actions taken:

Accepted as Tentative:

Tests for Automotive Hydraulic Brake Hose
Test for Compression-Deflection Characteristics of Vulcanized Rubber
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method at 80 C., as a tentative revision of the standard method
Spec. for Insulated Wire and Cable: Ozone-Resistant Insulation
Revisions of:
  Spec. for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 39 T)
  Spec. for Insulated Wire and Cable: Performance Rubber Compound (D 353 - 39 T)
  Spec. for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 - 39 T)
  Testing Flat Rubber Belting (D 378 - 39 T), with the following correction in the revised Section 15 (*a*):

Section 15. (*a*).—Change the next to the last sentence from the form as preprinted to read as follows by the addition of the italicized words:

Test specimens that break in the jaws or that, upon examination, are found to be imperfect shall be discarded, and retests shall be made *unless the results obtained in the first test exceed minimum values that may be required by detailed specifications.*

  Tests for Rubber Hose (D 380 - 39 T)
  Test for Compression Set of Vulcanized Rubber (D 395 - 37 T)
  Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 - 37 T)

Adopted as Standard:

Test for Abrasion Resistance of Rubber Compounds (D 394 - 37 T), as revised
Test for Accelerated Aging of Vulcanized Rubber (D 428 - 36 T), the two types of tests to be published as separate standards entitled "Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method," and "Test for Accelerated Aging of Vulcanized Rubber by the Oven Method"
Test for Flexing of Rubber Products (D 430 - 35 T), as revised
Air Pressure Heat Test of Vulcanized Rubber (D 454 - 37 T)
Spec. for Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 volts to Ground (D 120 - 37 T)

Papers:

The following papers were presented:

Accelerated Weathering of Transparent Plastics—G. M. Kline, W. A. Crouse and B. M. Axilrod, presented from manuscript by Mr. Kline.

The Absorption of Water by Plastics—G. M. Kline, A. R. Martin and W. A. Crouse, presented from manuscript by Mr. Kline.

Measuring the Plasticity of Molding Compounds—Robert Burns, presented by the author.

A Correlation of the Chemical and Electrical Changes Observed During the Oxidation of Mineral Insulating Oil—F. M. Clark, presented by the author.

The Standardization of Durometers—Lewis Larrick, presented by the author.

There being no further business the chairman then declared the Forty-third Annual Meeting adjourned *sine die.*

STANDARD SPECIFICATIONS FOR MATERIALS AND THEIR COMMERCIAL IMPORTANCE

ANNUAL ADDRESS BY THE PRESIDENT

H. H. MORGAN[1]

June 25, 1940

Before presenting my prepared address a few introductory remarks may be in order: All of the problems of our Society and our membership seem totally insignificant compared with the appalling events of the day. These events will have far-reaching and over-powering effects upon international affairs. With governments being destroyed, our whole civilization may change and our own living standards be vitally affected. Ahead of us is a national preparedness program that, in itself, is sure to affect our present living and that of generations to come. Experience has demonstrated and Colonel Young in his address on "Material Standards in National Preparedness"[2] has voiced the need of standardization to effect a state of preparedness sufficient to meet such emergencies as are conceivable. To do this well, to the extent necessary and as economically as possible, standardization of materials, fabrication and in general production-line methods must be instituted. It so happens, therefore, that Col. Young's subject and my own subject, Standard Specifications for Materials and Their Commercial Importance, are especially appropriate at this time. Standards in war preparedness and in commercial transactions are involved. The nearer such are to each other, just so much sooner will our United States be prepared and with the least ultimate burden on our citizenship.

Our Society is active. Our membership is essentially composed of men engaged in specification, inspection, testing and research work for the larger consumers of materials and the larger producers, and a considerable number having general interests. General interests include colleges, government and private research institutions, and a large number of companies engaged solely and independently in inspection, testing, and research.

Generally speaking, our Society activities are confined to materials and not final consumers' products. For example, we deal with specifications for boiler plates and boiler tubes but not with fabricated boilers; we deal with specifications for cement, sand, gravel, and stone but not with concrete structures. In general, the materials we concern ourselves with are either raw or semi-finished materials used in commercial trade.

In my remarks I shall not attempt to consider all our dealings with all, or any one group of materials, but will confine myself entirely to standard specifications used in engineering construction work because that has been the foundation of our recognized standing in our field.

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[1] Manager, Rail and Fastenings Dept., Robert W. Hunt Co., Chicago, Ill.

[2] See p. 3. This address was published in the A.S.T.M. BULLETIN, No. 105, August, 1940, p. 17.

I should like to have it clearly understood that I am not discussing specifications for equipment, which usually contain fabrication details of machinery, devices, or structures. Such specifications are special to the particular equipment or structure and involve matters of design that are necessarily a development of the engineer undertaking such work. However, it must be recognized that such specifications necessarily include material specifications in order that the quality of material for the various component parts is properly covered.

I wish, further, to emphasize that I am not discussing project specifications, which are complicated specifications involving an assembly of various specifications for equipment, devices and structures, and including legal and contract matters all requiring attention special to the particular project. Such project specifications necessarily involve the services of a special engineer, a structural engineer, or an architect.

However, such project specifications necessarily include standard specifications. This, of course, is to secure assurance that the materials used are of the proper standards. The specifications that are used should be those that are standards of commercial trade.

With this brief introduction, I will proceed.

The important specifications are standards of trade. Standardization has been the bedrock of civilization. The origin of standardization goes back to the crude beginning of human culture. The earliest records show a knowledge that there were standards of weights and measures of ancient Babylonia and Egypt. The Ten Commandments are basic standards of Christian living. Barter and trade early developed a standard basis of exchange or money. The development of these early standards to the present methods of effecting commercial transactions and the use of standard specifications is a history of interest that I cannot cover here.

I shall confine myself to the present status of standards in their application to specifications for materials and to engineering construction materials alone.

America is the land of the Biggest, the Best, and the Most. We have the biggest buildings, the best mass production methods, and the most money. These are the objectives of a new, virile, and energetic country, and we are that. We play hard and we work hard. When something appeals to us that is worth while, we take it seriously and we may "wind up" by overdoing it. This overdoing can apply even to the making of specifications.

Most specifications have their good points—which largely accounts for their present multiplicity. On the other hand, many specifications with good points also have their bad points, and these bad points naturally result in such specifications not being used and they die a natural death. A specification must have good or important points and must be free from bad points or unimportant requirements.

The subject is not a new one. It was discussed by Charles B. Dudley in his Presidential Address presented before our Society in 1903, in order to crystallize into definite statements the requirements for good specifications. So well did he accomplish his task that he became recognized as "the father of specifications." Since his day, so many have contributed their ideas that it would obviously be impossible to list and give credit to each. A few, however, may be mentioned: N. F. Harriman, Dean Harvey, J. W. Bancker, and Edward T. Gushee. Their contributions have been outstanding—but it should not be thought that they stand alone and unequalled.

Many times in recent years, discussions have been held on this matter of standard specifications of importance; but, generally, specifications for devices, equipment and projects have been involved and that has lead to confusion—sometimes hopeless confusion.

In this presentation I am trying to avoid the pitfalls of straying from the subject of my discussion which pertains essentially to standard specifications only and their commercial importance.

The three significant words in the title are (1) commercial, (2) specifications, and (3) materials. "Commercial" means in commercial use which means useful in commercial trade. By "specifications" is meant a definition of the buyers' requirements as an adjunct to the purchase order. "Materials" include only the fundamental material from which capital and consumer goods such as tools, devices, structures, or machinery are created. The title now takes on a definite and limited meaning. It says, essentially, "The importance of having, as an adjunct to a purchase order for the fundamental materials of industry, a definition of the buyers' requirements which is useful in commercial trade."

We must not overlook the fact that nearly all manufacturers of devices, appliances, or equipment are buyers of material. It is also true that the manufacturers of materials are heavy buyers of materials they themselves do not manufacture. The term "buyer" used in this discussion, therefore, applies to every company, corporation, or industry.

Standard specifications may be the specifications of an individual buyer, or a group of buyers of one industry, of a trade association, of a technical society—or may be the national joint standard of all industries buying or making the material. The specifications developed by the American Society for Testing Materials are of especial importance as they represent the product or development effected by the joint efforts of technical men engaged in the direct interests of both consumers or buyers and producers or manufacturers.

Specifications have a general or indirect importance, as well as a specific or direct importance. Indirectly, they tend to separate the significant from the insignificant, the tangible from the intangible, and to provide a common meeting ground for both buyer and seller. Directly, they furnish an efficient and economical basis upon which to deal with materials.

When we speak of the general benefits which accrue through the use of specifications, we mean *good* specifications. Poorly drawn specifications will not, for instance, distinguish between the significant items and those which have no significance. The more time one puts into the preparation of specifications the more value one gets out of them.

The same remarks apply to the separation of measurable qualities from those which are intangible. Materials are possessed of both qualities, but only those which can in some way be measured are of any value in a specification. Likewise, in providing a means by which the buyer and seller may speak the same language, commerce between the two is greatly facilitated, and the way opened for the more specific and direct benefits which a good specification, properly used, might bring to both.

These specific advantages of using good specifications may be listed as follows—(1) they enable the buyer to get what he wants; (2) the material is of uniform quality; (3) the buyer received goods more quickly and with less trouble; (4) he has access to wider markets; (5) costs are lower; (6) a suitable acceptance basis is established; (7) material becomes standardized, and (8) research is promoted.

The prime purpose of every specification, whether it be a national standard or that of a single individual, is to enable the buyer to tell the seller precisely what he wants—and then get it. It is for this one reason, and none other, that all specifications are written. It can readily be seen that this is of benefit to both buyer and seller and that the importance of this aspect cannot be overemphasized.

Not only does the buyer want material of certain qualities, he also wants to be sure of continually receiving goods of uniform quality. Generally his plans are laid and his operations set to work on material of unvarying character. Through specifications, the buyer has a control over his material, and by eliminating trouble from this source, his manufacturing processes or other application or use of material may proceed according to a predetermined schedule The supplier on the other hand, assured of a definite demand, may stabilize his operations for an even flow of production. From this follows, in logical order, the third advantage mentioned above—the least delay in securing materials.

Specifications which adequately tell the manufacturer what is wanted, and enable him to take proper steps to supply the demand, avoid many misunderstandings due to inaccurate and inadequate information, which result in delays due to correspondence or discussion to settle the issue. Material that is unsatisfactory and must be rejected upon receipt is especially expensive, for although the cost is not entered in the company's journal, it exists in the form of delayed production which may amount to more than the original cost of the material itself. One's ability to secure goods of the right quality without delay depends not only upon his being able to supply essential information to a certain supplier, but also in being able to choose between a number of manufacturers, scattered perhaps throughout the country. The possibility of choosing from a wide market is still another advantage.

In providing a wide choice of manufacturers from which the prospective purchaser may select one or more to fill his needs, one of the first services rendered is that of sweeping away trade names and other unsatisfactory restrictions. By devices such as trade names the buyer is limited by what he knows from experience about a few products; with his choice thus narrowed, he is at the mercy of a few producers. Furthermore, it may be an economic necessity to buy goods from Coast to Coast when plants are scattered throughout the country. In order to save freight rates and delays, it will be necessary for each unit to purchase from those suppliers in its immediate neighborhood. Yet each of the buyer's shops is designed to turn out exactly the same product and, consequently, must be supplied with exactly equivalent material. Specifications assure this wide market of equivalent material.

They go further; they assure it at the lowest possible cost consistent with the quality demand. This possibility of securing low prices is, therefore, another item of commercial importance inherent in the use of specifications. Because the supplier knows what is demanded of him, he can avoid trial-and-error methods in order to meet the buyer's demands. This fact, combined with the consequent stabilization of product and producing processes, makes it possible to offer lower prices to the buyer. Not only are actual costs reduced in the manufacture of goods and these savings passed on to the consumer, but the purchaser has access to a wide and hence competitive market. Competing manufacturers are asked to supply equivalent values at prices reflecting their varying degrees of skill and initiative.

The suppliers, in their eagerness to obtain an order, often overestimate their ability to produce goods of a certain quality at the price quoted. Unless specifications are used as a basis of acceptance, the buyer may receive goods of inferior quality from a producer who has over-reached himself.

This provision of a suitable basis upon which to accept goods is another very useful adjunct of specifications. By stating exactly what testing or analysis is required and to what extent inspection will take place, the producer knows beforehand the standards his product must meet in order to be accepted. The provision for uniform inspection, testing, and analysis does away with discrepancies which might and often do cause controversies.

A specification which by wide use and agreement has become a national standard is of more importance than one set up by a single buyer. Material manufactured to such a national specification becomes a standard product, known throughout the industries. Manufacturers are prepared to supply it in the most efficient and economical manner, while those who buy know from experience what to expect of the material—its potentialities and limitations—and have built their own product around the uniform qualities of the material. The advantages of a wide market, uniform product, quicker and easier deliveries, and lower costs are all open to the user of a national standard.

And yet, new specifications by individual buyers are becoming more and more numerous. This is essentially due to the thought that a special use or application justifies certain specialized requirements. The buyer may or may not be justified, but I am inclined to believe that too often he has not recognized the undoubted fact that a material conforming to a specification already written would be satisfactory for his use with only minor supplemental restrictions on such items as tolerance, workmanship, or finish.

Quite apart from the direct benefits of specifications as just noted, and yet intimately tied up with their use, is their value in promoting research. The buyer (in making a new specification or in considering the value of a national standard) and the seller (attempting to meet the provisions and limitations placed upon his product in the most efficient manner) will both be led to examine critically the product and its possibilities. I will repeat that the buyer and the seller will both be led to examine critically the product and its possibilities. This is research and may go only so far as to develop efficiency in handling the product, or it may develop new uses and potentialities hitherto unsuspected. It may even lead to the rejection of one material in favor of another. Regardless of the exact outcome, benefits of a permanent nature are sure to result from such investigation.

While this discussion is concerned only with the commercial importance of specifications in use and not with their preparation, yet in order to secure the potential advantages they must be properly drawn. A few words on that subject may not be amiss.

Above all, specifications should be clear and complete. Ambiguities of meaning or lack of vital requirements or instructions will rob the specification of all its value. On the other hand, severe restrictions should be avoided; limitations, while they may come from chemical analysis, physical tests, microscopic examination, or methods of manufacture, should be governed strictly by the service desired. It is *service* that the buyer wants—not a mass of physical or chemical data.

Clearness, completeness and sane restrictions can be attained only through the joint efforts of buyer and seller. The material has two aspects, both of which must be taken into account—first, the problems of its manufacture; second, its use by the purchaser. Without a clear understanding of both sides, gained only through the cooperation of producer and consumer, incongruities will quite likely appear to wreck all the fondest hopes of the specification writer.

Due to the steady progress of industry, a specification, however well written at the time, cannot stand long without occasional revision. New products and improved methods of manufacture constantly tend to change the original conditions. Above all, therefore, the specification should be open to revision.

Not only should the specification describe exactly what is wanted, but such qualities as are desired should be subject to tests, analyses, and inspection. When these are well known, mention alone is all that is needed, but when new methods are used or different results are expected, adequate descriptions should be included. Inspection by the buyer or his representative is essential if he is to be assured that the tests and analyses are impartially made.

The extent to which material should be subjected to inspection should be clearly stated, and this, in turn, depends largely on its importance. If the material is ordered in large quantities or if its failure in service would mean costly delays in production or loss of life or money, then constant and rigid inspection should be employed. Wherever practicable this should be performed at the point of manufacture. On relatively unimportant material bought in small quantities, less frequent inspection is needed.

To sum up: The practical importance of specifications hinges directly on their ability to assure the buyer he is receiving exactly the goods he orders. In the competition of trade, particularly in regard to heavy products under mass production, it is essential to know that materials are furnished according to specifications. Adequate specifications for materials with competent inspection assure to the buyer a maximum usefulness and serviceability of material at a minimum of cost. The efficiency of any buying organization is measured thereby.

While a large percentage of buyers cannot maintain the expert personnel or research facilities to write adequate specifications, all want to increase their efficiency. They can do this by utilizing standard specifications, materials, and the facilities afforded by research institutions as well as by using commercial inspection and testing laboratories organized for this purpose.

Standard specifications are prepared to establish quality tolerances and test and inspection procedure on a material which has a wide application in the fabrication of various devices or equipment items. Whether such standard materials, under special heat treatment or fabrication processes, can be used to advantage in any particular device or piece of equipment is a matter of research development on the part of the buyer. However, the consumer must cooperate with the producer in establishing a basis of specification requirements that will comprehensively fix the significant qualities, both physical and chemical, of the material, the mechanical tolerances, and the test and inspection procedure so that he may know a delivery to the specifications will have certain properties and a uniformity in properties that will fit or adapt itself to the fabricating and treatment practice necessary in the manufacture of the device, equipment, or structure.

It is just in this manner that the specifications of the A.S.T.M. are developed. Consequently, they represent a character of specifications that has the maximum of commercial importance. In my intimate contact with the activities of the A.S.T.M. over practically twenty-five years, I have been, perhaps, in preferred position to observe how significant features of specifications under development by them have been the subject of discussion, investigation and ultimate solution along practical lines that have resulted in useful specifications. That the A.S.T.M. specifications are almost universally used in the large field of engineering construction materials, is evidence of their commercial importance.

In closing, I hope that I have given you some conception of what makes a standard specification for material of commercial importance, and why we are all interested and participate in the strenuous activities of the A.S.T.M.

ANNUAL REPORT OF THE EXECUTIVE COMMITTEE

The Executive Committee offers the following report on general Society activities for the year ending with this annual meeting:

Membership:

The membership of the Society on June 1, 1940, was 4314. Statistics showing gains and losses in various classes of membership are as follows:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1939	June 1, 1940	Resignations	Dropped	Deaths	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	6	6	...	...	...	...	...	...	...	...	...
Life or Perpetuity	10	10	...	...	...	...	...	...	...	...	...
Sustaining	21	88	...	...	...	...	58	9	...	67	67
Company, Firm, etc.	1022	1007	27	6	...	61	6	73	94	79	15[a]
Individual, etc.	3090	3140	127	53	21	6	12	245	207	257	50
Junior	50	63	3	6	...	9	...	31	18	31	13
Total	4199	4314	157	65	21	76	76	358	319	434	115
Student	356	411	30	173	...	12	...	270	215	270	55

[a] Loss.

The net gain for the year of 115 compares with 43 for the preceding year and 164 for two years ago. Elections to membership were 358 compared to 303 for the preceding year; losses from resignations are slightly lower and losses from delinquencies slightly higher than for the preceding year. The number of student members increased from 356 to 411.

An important accomplishment of the year is the very substantial increase of 67 in the number of sustaining members, bringing the total to 88. The names of these new sustaining members listed in the order of acquiring membership are:

Standard Oil Co. (Ohio)
Ethyl Gasoline Corp.
International Harvester Co.
Consolidated Edison Co. of New York, Inc.
Robert W. Hunt Co.
Indiana Steel and Wire Co.
Standard Oil Co. of California
Commonwealth Edison Co.
The Firestone Tire and Rubber Co., Akron, Ohio.
Climax Molybdenum Co.
Louisville Cement Corp.
Ajax Metal Co.
Bethlehem Steel Co., Inc.
Public Service Electric and Gas Co.
Standard Oil Development Co.
Owens-Illinois Glass Co.
New Jersey Zinc Co.
American Steel Foundries
E. I. du Pont de Nemours and Co., Inc.
Youngstown Sheet and Tube Co.
Union Carbide and Carbon Research Laboratories
Doehler Die Casting Co.
National Tube Co.
The Pure Oil Co.
Reichhold Chemicals, Inc.
United States Rubber Co.
Standard Oil Co. (Indiana)
Sears, Roebuck and Co., Technical Laboratories, Department 817
The Commonwealth and Southern Corp.
The Clark Thread Co.
American Viscose Corp.
Cleveland Electric Illuminating Co.
Philadelphia Electric Co.

Wellington Sears Co.
Celanese Corporation of America
Hydro-Electric Power Commission of Ontario
American Smelting and Refining Co.
American Car and Foundry Co.
A. P. Green Fire Brick Co.
Potomac Electric Power Co.
Sargent & Lundy, Inc.
Arlington Mills
American Steel and Wire Co.
Taylor Forge and Pipe Works
Pittsburgh Steel Co.
Shell Development Co.
Goodyear Tire and Rubber Co.
Missouri Portland Cement Co.
Copperweld Steel Co.
Sun Shipbuilding and Dry Dock Co.
American Creosoting Co.
The Duriron Co., Inc.
A. O. Smith Corp.
Camden Forge Co.
Chicago Bridge and Iron Co.
Caterpillar Tractor Co.
Marquette Cement Mfg. Co.
Plaskon Co., Inc.
Duke Power Co.
Union Oil Co. of California
Forstmann Woolen Co.
Apex Smelting Co.
Leeds & Northrup Co.
The Titanium Alloy Manufacturing Co.
St. Joseph Lead Co.
Minnesota Mining and Mfg. Co.
American Bureau of Shipping

Of these, 58 were acquired by transfer of existing company memberships (which accounts for the net decrease in the number of company members through the year) and nine were acquired by companies which had not previously held company membership, although most of them had been affiliated with the Society's activities through one or more individual memberships. The gratifying response of so many companies to the invitation to lend increased financial support to the Society's activities through this form of membership is greatly appreciated by the Executive Committee. Many other companies are seriously considering this form of membership and it is hoped the number will further increase during the coming year.

The substantial growth of Society membership is due in considerable measure to the efforts of many members of the Society and the several district committees who have given unstintingly their help and cooperation in this important work, for which the Executive Committee expresses its thanks.

The Society has lost the following members through death:

	Date of Membership
Angus, James	1929
Boylston, H. M	1907
Bregowsky, Ivan M	1904
DeBerlhe, A. Berlhe	1921
Durkee, Frank W	1926
Fromlet, Victor H	1933
Gilpin, Francis H	1928
Hanks, Abbot A	1917
Hughes, Edward E	1912
Hurlburt, Edward N	1922
Lang, P. G., Jr	1939
MacPherran, R. S	1902
Marshall, Emery C	1938
McCorkle, I. B	1928
McKinley, J. M	1917
Merriman, Thaddeus	1922
Mussey, William H	1912
Norton, Charles L	1903
Penniman, W. B. D	1927
Setzler, H. B	1926
Spicer, Clarence W	1925
Wells, A. P	1919
Willman, Eugene C	1919

Many of these members were engaged in the technical and administrative work of the Society, among them H. M. Boylston, former chairman of Committee A-4 on Heat Treatment of Iron and Steel, I. M. Bregowsky, Abbot A. Hanks, former chairman of the Northern California District Committee, E. E. Hughes, E. N. Hurlburt, R. S. MacPherran, former chairman of Committee A-3 on Cast Iron, J. M. McKinley and Thaddeus Merriman.

Considerable emphasis is being placed by the Membership Committee on the advantages of A.S.T.M. membership to junior engineers. An editorial by J. R. Townsend, chairman of the Membership Committee, presenting these advantages was published in the October, 1939, issue of the ASTM BULLETIN. This statement was brought directly to the attention of the officers of each standing committee with a letter recommending that the members of these committees endeavor to bring or send younger members of their staff to the Society's meetings, arranging where possible for some specific work or responsibility to be assumed by these younger men, which should lead to important benefits not only to the junior engineer but also to the Society. The following quotation from the above-mentioned letter gives concisely the reasons why the Executive Committee considers this subject to be worthy of the serious consideration of all members of the Society:

> "We feel that the A.S.T.M. will best grow and fulfill its functions of preparing timely specifications and test methods if there is a constant infiltration of young men into the Society's work. The A.S.T.M. provides a unique opportunity for young technologists to work with those more experienced on specific projects. This permits the younger engineers to acquire the art of those more experienced and to broaden themselves in the direction of dealing with other people. The necessity to negotiate and compromise, to cooperate, and to combine ideas of many people has always been and will ever continue to be one of the prime functions of engineering. An unusual opportunity is provided by the A.S.T.M. for training in this direction."

It will be recalled that two years ago the Executive Committee instituted the award of certificates commemorating forty years of continuous membership in the Society. Eighteen such awards have been made in the past two years, and this year the following eight members who have completed forty years of continuous membership will be awarded certificates at this annual meeting:

F. H. Clark
Theodore L. Condron
Robert Job
Charles B. Wing
American Steel and Wire Co.
Colorado Fuel and Iron Co.
National Tube Co.
John A. Roebling's Sons Co.

Committee Activities:

Despite the extensive committee work that preceded the last annual meeting in preparation for the revised 1939 edition of the Book of Standards, the work of the committees during this past year, as recorded in their annual reports at this meeting, has been unusually active. Many new standards are being proposed, including a number in some of the newer fields in which the Society is now engaged.

The current reports of Committee E-9 on Research and Committee E-10 on Standards deal with certain phases of the committee work and, supplemented by reviews that will appear in the August and October issues of the ASTM BULLETIN, give a general summary of the committees' activities. This report, therefore, makes brief reference only to several administrative actions during the year.

The annual meeting last year referred to the Executive Committee certain recommendations regarding the publication of proposed Specifications for Emulsion for Use in Densely Graded Cold Asphaltic Mixes, as well as publication of certain test methods related thereto, which were to be balloted upon by Committee D-4 following the annual meeting. The Executive Committee, after reviewing the results of the letter ballot, decided that publication of these specifications should be deferred pending opportunity for further consideration of Committee D-4 during the year, and that the proposed methods of testing such emulsions be published in the ASTM BULLETIN as information. Accordingly these methods appeared in the December, 1939, issue.

The Executive Committee gave consideration to the following resolution adopted during the 1939 annual meeting at a Round Table Discussion on Freez- and Thawing Tests:

> "That a committee be organized under the proper auspices of the Society, as for example Committee E-1 on Methods of Testing, to continue studies of freezing and thawing tests looking towards the establishment of a suitable standard procedure, with the understanding that this committee would act as a clearing house for information on freezing and thawing tests and serve in an advisory capacity to the interested standing committees."

Since it appeared from discussion of this resolution that what may really be required is a fundamental study of the phenomena of freezing and thawing and of the property of durability in general, looking to the establishment of basic principles upon which standard procedure may ultimately be based, it was felt that the whole subject should be considered in the nature of a research rather than a standardization project. Accordingly this subject was referred to Committee E-9 on Research for further study and suitable action.

During the year the Executive Committee received a recommendation from Committee E-9 on Research that there be formed in the Society either a separate standing committee on Hydrogen Ion Determinations or a new technical committee of Committee E-1 on Methods of Testing on this subject. Committee E-9 had reached the conclusion that it was inadvisable for the Society to undertake any research work on this subject but that there is a need in the Society for studying the standardization of methods of determining hydrogen ion concentration in various materials. Accordingly Committee E-1 was requested to consider the formation of a technical committee on this subject, to act in an advisory capacity to other standing committees requiring information and to develop such methods of test that might prove to be needed in the Society. After considering the matter Committee E-1 has agreed to undertake the formation of a new technical committee of Committee E-1 on Hydrogen Ion Determinations.

Upon its request Committee B-6 on Die-Cast Metals and Alloys has been authorized to solicit funds from producers and consumers of die castings in support of the committee's present program of research.

A proposal is under consideration for the formation of a separate standing committee in the Society to deal with the subject of specifications and tests for electrodeposited coatings on both ferrous and non-ferrous metals. This subject at present comes under the jurisdiction of three standing committees, namely, A-5 on Corrosion of Iron and Steel, B-3 on Corrosion of Non-Ferrous Metals and Alloys, and B-6 on Die-Cast Metals and Alloys, who together with the American Electro-Platers' Society and the National Bureau of Standards have formed joint committees that are dealing with phases of the matter that include both research and standardization activities. The organization setup for this important work seems to have become unduly complicated and the Executive Committee has requested the three standing committees named above to state whether they are prepared to relinquish their jurisdiction in the matter of standards for electrodeposited coatings to the proposed new standing committee. This method of handling the development of standards in this field is also being discussed with the American

Electro-Platers' Society and the National Bureau of Standards.

The Executive Committee has approved a recommendation of Committee D-11 on Rubber Products that its statement of scope be changed to read as follows so that the committee may include rubber-like products within its activities:

> "The formulation of specifications and methods of test for products made from rubber or rubber-like materials, including research investigations concerning the properties of these materials."

No change in the name of the committee is considered necessary.

The Executive Committee is giving consideration to certain questions of jurisdiction between Committees D-4 on Road and Paving Materials and D-18 on Soils for Engineering Purposes, having to do with the development of standards dealing with stabilization of soils for highway purposes.

The Executive Committee is undertaking to reorganize Committee C-18 on Natural Building Stones and Slate, which has been inactive for a considerable time. A committee on reorganization has been appointed consisting of W. C. Clark, Public Works Branch, Procurement Division, chairman, T. I. Coe, American Institute of Architects, and D. W. Kessler, National Bureau of Standards.

Administrative Regulations:

Jurisdiction over Regulations Governing the Form but not the Substance of Standards has been transferred from Committee E-5 on Standing Committees to Committee E-10 on Standards. The latter committee has extensively revised and condensed these regulations, which are now published under the title "Recommendations on the Form of Standards."[1] They are intended for the guidance of standing committees in the preparation of specifications and methods of test in order to simplify the task of establishing and maintaining a reasonable degree of uniformity in the Society's standards.

The Executive Committee has revised the Regulations Governing the Finance Committee[2] primarily to provide adequately for authority to invest the funds of the Society; to sell, assign or transfer securities; and to arrange for collateral loans. A few minor revisions have been made, including a modified and more suitable requirement for obtaining competitive bids for printing the Society publications. The revised Regulations will be published in the 1940 Year Book.

District Committees:

The district committees have done good work during the year in the interest of the Society through the holding of local meetings under their sponsorship, as noted later in this report, through activities in securing new members and in other ways. The Executive Committee wishes to express its appreciation to the officers and members of these committees for the time and thought that they are devoting to this work.

The fourth conference of officers of district committees was held during the Spring Meeting at Detroit in March, 1940, at which a number of matters of interest to the district committees were considered. Several features of the Spring Meetings held under the sponsorship of the district committees were discussed with considerable benefit. The plan adopted for the first time by the Detroit District Committee of defraying expenses of the Spring Meeting program was outlined, which in this instance has also made possible the addition of a substantial sum to the treasury of that committee. Various methods of handling membership promotion activities were considered, including a scheme that the Chicago District Committee is trying out of offering a copy of one of the Society's compilations of standards or other suitable publications to persons who secure new members.

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[1] See 1939 Year Book, pp. 293 to 298.

[2] See 1939 Year Book, pp. 276 to 277.

Meetings:

The various meetings held during the past year have been fully reported in the ASTM BULLETIN so it is sufficient here simply to cite them as a matter of official record, with an expression of appreciation from the Executive Committee to the many members of the committees who helped to make these meetings successful.

The eleventh Spring Meeting and meetings of committees were held at Detroit, March 4 to 8, 1940, and featured a Symposium on New Materials in Transportation. The total registration was 460, with upwards of 110 committee, subcommittee, and section meetings. Plans for the meeting were developed by the Detroit District Committee under the chairmanship of T. A. Boyd, with the able assistance of the entire committee.

The following local meetings were held under the auspices of the respective district committees: In St. Louis on November 27, 1939; in Chicago, November 28, 1939, and March 27, 1940; in Pittsburgh, January 29, 1940; in New York, December 12, 1939, and April 4, 1940; in Los Angeles, May 10, 1940. Accounts of each of these meetings have appeared or will appear in the ASTM BULLETIN. President Morgan attended the meetings in St. Louis, Chicago and the first meeting in New York and conveyed appropriate greetings from the Society to the members and guests present. In the unavoidable absence of the President, J. J. Shuman and Dean Harvey conveyed the President's greetings at the Pittsburgh meeting.

The Executive Committee announces that the next Spring Meeting and group committee meetings will be held in Washington, D. C., March 3 to 7, 1941. The next Annual Meeting, together with the Sixth Exhibit of Testing Apparatus and Related Equipment, will be held in Chicago, Ill., June 23 to 27, 1941.

Publications:

This has been a busy and eventful publication year. How busy it was may be seen from the information concerning publications that follow; and it was eventful because of the revised edition of the Book of Standards published in the new double-column format in three parts: I, Metals; II, Nonmetallic Materials, Constructional; III, Nonmetallic Materials, General; and including for the first time the tentative standards and tentative revisions of standards. Omission of the latter from the *Proceedings* has made possible the publication of the *Proceedings* in a single volume containing committee reports, technical papers, and discussion. The Book of Tentative Standards is no longer published. The new Book of Standards has been favorably received by the members and throughout the industries and both the Executive Committee and Committee E-6 on Papers and Publications, under whose direction all publications are edited and issued, feel that a long step forward has been taken in increasing the usefulness of the Society's standards.

The following regular publications were issued and distributed to the members:

1939 Proceedings: This year, for the first time since 1921, in one part, comprising 1352 pages, 4850 copies. In addition, preprints of reports and papers totaled 1355 pages, 1500 copies.

1939 Book of Standards:

Part I, Metals, 1344 pages, 8600 copies.
Part II, Nonmetallic Materials, Constructional, 1249 pages, 6700 copies.
Part III, Nonmetallic Materials, General, 1201 pages, 6500 copies.

A.S.T.M. Methods of Chemical Analysis of Metals, 256 pages, 3200 copies (intended as a two-year supply).

1940 Index to A.S.T.M. Standards, Including Tentative Standards, 160 pages, 16,500 copies.

Year Book, 304 pages, 3200 copies.

ASTM BULLETIN, six issues, August 1939 to May 1940, 360 pages; average edition, 6530 copies.

The following symposiums held at the Columbus Regional Meeting in 1939 were published and furnished to members on order:

Symposium on Thermal Insulating Materials, 127 pages, 2000 copies.

Symposium on Lime, 124 pages, 2000 copies.

A statement of the volume of the above-mentioned publications for the past four years follows. This statement is not truly comparative because all the standards published in 1936 and 1937 and most of those in 1938 were in the single-column format that has now been discarded.

Publication	1936, pages	1937, pages	1938, pages	1939, pages
Proceedings	2111	2092	2084	1352
Book of A.S.T.M. Standards	2419	....	....	3794
Supplement to Book of A.S.T.M. Standards	....	282	251	....
A.S.T.M. Methods of Chemical Analysis of Metals	260	260	35	256
Index to A.S.T.M. Standards and Tentative Standards	160	120	128	160
Year Book	246	272	286	304
Book of A.S.T.M. Tentative Standards	1422	1664	1796	....
Regional Meeting Symposiums	132	232	57	251
	6750	4922	4637	6117

During the year the Society also published the following compilations of standards:

A.S.T.M. Standards on Cement (Committee C-1), 103 pages, 2000 copies, published annually.

A.S.T.M. Standards on Petroleum Products and Lubricants (Committee D-2), 348 pages, 4900 copies, published annually.

A.S.T.M. Standards on Electrical Insulating Materials (Committee D-9), 319 pages, 700 copies, published annually.

A.S.T.M. Standards on Rubber Products (Committee D-11), 218 pages, 1200 copies, published biennially.

A.S.T.M. Standards on Textile Materials (Committee D-13), 336 pages, 1300 copies, published annually.

A.S.T.M. Specifications for Pipe and Piping Materials for High-Temperature and High-Pressure Services (Committee A-1), 143 pages, 1500 copies, published annually.

Selected A.S.T.M. Standards for Students in Engineering, 224 pages, 6000 copies.

The following four publications were also issued, the last three being reprints from the *Proceedings:*

Evaluation of Petroleum Products (Sectional Committee Z 11), 56 pages, 2500 copies.

Wire Test Report (Subcommittee VIII, Committee A-5), 117 pages, 1 large insert plate, 2000 copies.

Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, 18 pages, 5 large insert plates, 1000 copies.

Marburg Lecture: "Stress, Strain, and Structural Damage," by H. F. Moore, 26 pages, 3000 copies.

Charge for Right to Reprint Standards.—The Executive Committee has established the policy of making a charge for the right to reprint the copyrighted A.S.T.M. standards and tentative standards. For many years no charge for this right has been made. In recent years many organizations have for excellent reasons found it desirable, in their utilization of A.S.T.M. standards, to reprint them in their own type format for distribution instead of purchasing them from the Society, which in general is preferred and is usually done. Thus the Society is deprived of a needed source of income from sales of publications, to an extent which has come to be quite considerable with the rapidly expanding use of A.S.T.M. standards.

It is equitable that the Society should be reimbursed for this privilege, inasmuch as all income from publication sales is applied to the operations of the Society which benefit not only its members but industry generally.

Accordingly, a scale of charges for the right to reprint standards and tentative standards has been adopted, the charges depending upon the number of standards and the number of copies to be printed. Anyone desiring to reprint one or more A.S.T.M. standards or tentative standards should communicate with the Secretary-Treasurer of the Society for the required permission, whereupon the charges applicable to each case will be quoted.

The charges apply to all reprinting in full, either as separates or in pamphlet form, or as part of a book. No charge is made for reprinting excerpts from standards, although permission for excerpting should be obtained. It is required that suitable acknowledgment be made to the Society in all cases.

Finances:

Report for the Fiscal Year 1939.—The annual statement of the finances of the Society follows in the report of the auditors for the fiscal year, January 1 to December 31, 1939. The report gives the balance sheet of assets and liabilities, including special and designated funds (that is, the Research Fund, Medal and Lecture Fund, and Committee Funds); statement of receipts and disbursements, in which items are classified into "operating" (budgeted) and "nonoperating"; details of special funds; and investment of Society funds, both book and market values being given.

Total receipts from dues and entrance fees in 1939 were $82,348.69, an increase of about $1800 over corresponding receipts for the preceding year. Receipts from sales of publications totaled $51,640.05, being the second highest figure in the Society's history. The percentage ratio to total receipts is 34.4 per cent, which is about average for recent years. The fact that one-third of the Society's income is derived from sale of its publications is significant in showing the Society's dependence on this source of income as a means of carrying on its work. Miscellaneous receipts amounted to $16,178.27, consisting principally of receipts from advertising in the ASTM BULLETIN and the Index to Standards and Tentative Standards, receipts from the Exhibit of Testing Apparatus and Equipment, interest and dividends, and registration fees. Advertising in both BULLETIN and Index was slightly greater than the preceding year.

Total operating receipts were $150,167.01. Total operating disbursements with all current bills paid, as detailed in the auditors' report, were $151,685.97. The resulting deficit of $1518.96 was met from reserve acquired from operations of the fiscal year 1937.

Because of factors introduced by the triennial publication of the Book of Standards, current operations of the Society must be reviewed over a three-year period for a true picture. For the three-year period comprising the fiscal years 1937, 1938, and 1939 there is a favorable balance between operating receipts and operating disbursements of $10,679.16.

In the statement of liabilities, attention is called to the fact that there is still included the Book of Standards' Reserve of $12,000. Normally this reserve is completely expended in the year in which the triennial Book of Standards is published. However it was not possible to complete Parts I and II of the Book in 1939; accordingly only a portion of the costs of the complete Book of Standards was placed on the books

REPORT OF THE AUDITORS FOR THE FISCAL YEAR, JANUARY 1, TO DECEMBER 1, 1939

Philadelphia, January 11, 1940.

MR. C. L. WARWICK, *Secretary-Treasurer,*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of your Society for the year ended December 31, 1939. We did not make a detailed audit of all transactions, but made extensive test checks to determine the accuracy of your records.

We have prepared and submit herewith Balance Sheet as of December 31, 1939, Statement of Cash Receipts and Disbursements for the year ended that date and other supporting schedules, all of which are in agreement with the records of your Society. In our opinion, these statements present fairly the financial position of the Society at December 31, 1939, and the results of its operations for the year then ended.

The investments owned by the Society as detailed were examined by us, with the exception of $17,000 Baltimore & Ohio R. R. Co. Gen & Ref Bonds which were deposited with the Girard Trust Co. under plan of modification of interest charges and maturities and for which a receipt of said depository was submitted.

All income from investments was properly accounted for. In this connection the income from 98 shares of General Motors Co. stock is not included in the income of the Society in any of the schedules herewith, as same is payable to an annuitant as and when received under the terms of an agreement dated February 20, 1937.

Respectfully submitted,

(Signed) JOHN HEINS & CO.
Certified Public Accountants

BALANCE SHEET AS OF DECEMBER 31, 1939

(Including Special and Designated Funds)

ASSETS

General Funds:			
Current: Cash	$30 029.65		
Less checks drawn as of December 31, 1939—(Cost of Proceedings $10 200 and Book of Standards and Tentative Standards $10 500) in hands of Secretary-Treasurer that date	20 700.00		
		$9 329.65	
Investments (Market Value, $47 961.39)—Cost		54 902.10	
Accounts Receivable		13 939.06	
Total Current Assets—General Fund			$78 170.81
Furniture and Fixtures			6 467.27
A.S.T.M.-A.S.M.E. Symposium on Effect of Temperature: Special Account for Financing Book			941.47
Total General Funds			$85 579.55
Special and Designated Funds:			
Cash: A.S.T.M. Research Fund		2 142.55	
Dudley Medal and Marburg Lecture Fund		518.63	
Committee Funds		9 508.26	
			12 169.44

Investments:			
A.S.T.M. Research Fund (Market Value, $18 873.26)—Cost or book value	22 890.86		
Dudley Medal and Marburg Lecture Fund (Market Value, $1 892.50)—Cost	6 625.00		
Committee Funds (Market Value, $6 535.00)—Cost	6 000.00		
			35 515.86
Total Special and Designated Funds			$47 685.30
Total Assets			$133 264.85

LIABILITIES

General Funds:			
Current: Accounts Payable:			
Committee A-1	$3.86		
Committee C-1	259.79		
Advance Collections, Members and Publications	2 513.36		
Total Current Liabilities—General Fund		$2 777.01	
Life Membership Fund	$3 005.20		
Headquarters Fund	2 564.48		
Publication Fund	3 015.00		
A.S.T.M.-A.S.M.E. Creep Data Volume	1 100.72		
Book of Standards Reserve	12 000.00		
Book of Standards Reserve, Account of Sales for Future Delivery	3 000.00		
Surplus	58 117.14		
		82 802.54	
Total General Funds			$85 579.55
Special and Designated Funds:			
A.S.T.M. Research Fund		$25 033.41	
Dudley Medal and Marburg Lecture Fund		7 143.63	
Committee Funds		15 508.23	
Total Special and Designated Funds			47 685.30
Total Liabilities and Funds			$133 264.85

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1935–1939, INCLUSIVE

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable and Miscellaneous	Furniture and Fixtures	Accounts Payable and Miscellaneous	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1935	$10 514.73	36 180.42	9 220.39	7 489.70	2 695.60	10 023.44	7 061.76	43 624.44
1936	6 943.82	29 767.23	16 088.91	6 915.95	2 624.62	....	7 061.76	50 029.53
1937	20 686.08	37 599.49	10 506.78	7 029.87	2 140.86	6 000.00	9 061.76	58 619.60
1938	24 425.51	39 568.24	9 609.00	6 912.96	2 283.88	12 000.00	8 898.99	57 332.84
1939	9 329.65	54 902.10	14 880.53	6 467.27	5 777.01	12 000.00	9 685.40	58 117.14

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD JANUARY 1, 1939, TO DECEMBER 31, 1939

Cash balance, January 1, 1939			$24 425.51
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$77 696.61		
Past Dues	664.08		
Advance Dues	1 310.50		
Income, Life Membership Fund	150.00		
Entrance Fees	2 527.50		
Total Dues		$82 348.69	
Sale of Publications:			
Separate Standards	7 512.12		
Book of Standards	5 176.85		
Book of Standards (Members, addl. parts)	5 122.28		
Combined Book of Standards and Tentative Standards.	1 745.08		
Book of Tentative Standards	5 684.63		
Methods of Chemical Analysis of Metals	665.71		
Proceedings	4 867.05		
Selected Standards for Students in Engineering	827.00		
Bulletin Subscriptions (Non-Members)	252.40		
Special Reprints and Miscellaneous	19 786.93		
Total Sale of Publications		$51 640.05	
Miscellaneous:			
Binding (Members)	192.00		
Advertising, Bulletin	5 552.40		
Advertising, Index	1 725.00		
Interest and Dividends	2 876.61		
Registration and Other Fees, Annual Meeting	1 471.00		
A.S.T.M. Exhibit, Annual Meeting	3 430.75		
Committee A-1 for Technical Assistant	868.93		
Miscellaneous	61.58		
Total Miscellaneous Items		$16 178.27	
Total Operating Receipts (Budgeted)		$150 167.01	
Nonoperating Receipts (Not Budgeted):			
Investments, Matured or Sold	$17 552.79		
Excess Remittances	237.42		
Accrued Interest	17.08		
Creep Data Volume	982.56		
Symposium on Effect of Temperature	147.70		
Committee C-1 for Technical Assistant	1 650.00		
Total Nonoperating Receipts		$20 587.55	
Total Receipts			170 754.56
Carried Forward			$195 180.07

Brought forward			$195 180.07
DISBURSEMENTS			
Operating Disbursements (Budgeted):			
Publications:			
Year Book	$1 964.29		
Book of Standards	242.00		
Combined Book of Standards and Tentative Standards	16 979.17[a]		
Selected Standards for Students	943.24		
Methods of Chemical Analysis of Metals	1 158.39		
Proceedings	13 350.84[b]		
Preprints	3 116.00		
Regional Meeting Papers	1 001.10		
ASTM Bulletin	7 430.80		
Circulars to Members	1 239.80		
Separate Standards	3 401.01		
Index to Standards and Tentative Standards	2 013.17		
Reprints (Reports, Papers, Symposiums, etc.)	8 219.79		
Miscellaneous Printing (Authors' Reprints, etc.)	628.64		
Resetting Standards (Double Column)	3 988.09		
Total Disbursements, Publications		$65 676.33	
Salaries		54 672.12	
General Office Expenses		10 965.76	
Expenses, Standing and District Committees		3 688.29	
Expenses, Meetings and A.S.T.M. Exhibit		4 500.62	
Traveling Expenses, Administrative and Special Committees		2 076.32	
American Standards Assn. and Sectional Committees		1 000.00	
Rent, Headquarters		6 000.00	
Furniture and Fixtures		478.21	
Principal, A.S.T.M. Research Fund (one-half current year's entrance fees)		1 250.00	
Employees Retirement Fund		950.32	
Miscellaneous (Dues, Contributions, etc.)		428.00	
Total Operating Disbursements (Budgeted)		$151 685.97	
Nonoperating Disbursements (Not Budgeted):			
Investments	$31 407.25		
Accrued Interest	19.58		
Refund of Excess Remittances	237.42		
Purchase of Retirement Income	750.00		
Symposium on Effect of Temperature	4.11		
Creep Data Volume	157.93		
Technical Assistant, Committee C-1	1 588.16		
Total Nonoperating Disbursements		34 164.45	
Total Disbursements			$185 850.42
Cash Balance, December 31, 1939			$9 329.65

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[a] This amount includes $10,500.00 covering checks drawn against cost of Book of Standards and Tentative Standards, but not actually paid on December 31, 1939, pending final completion of the publication work.

[b] This amount includes $10,200.00 covering checks drawn against cost of *Proceedings*, but not actually paid on December 31, 1939, pending final completion of the publication work.

In addition to the general funds of the Society, the Secretary-Treasurer has on hand the following funds:

A.S.T.M. RESEARCH FUND

Principal Account:	*Total*	*Invested*	*Uninvested Cash*
Balance, January 1, 1939	$22 878.18	$21 660.07	$1 218.11
Receipts—Sale of Securities		−2 490.00	+2 644.50
Profit on Sale of Securities	+154.50		
Contribution — A.S.T.M. Entrance Fees	+1 250.00		+1 250.00
	$24 282.68	$19 170.07	$5 112.61
Disbursements—Purchase of Securities	....	+3 720.79	−3 720.79
Balance, December 31, 1939	$24 282.68	$22 890.86	$1 391.82
Income Account:			
Cash Balance, January 1, 1939	$450.42		
Receipts—Interest and Dividends	699.70	$1 150.12	
Disbursements:			
Fiber Board Investigation (Committee D-6)	24.39		
Painting of Structional Iron and Steel (Committee D-1)	75.00		
Speed of Testing Metals (Committee E-1)	300.00	399.39	
Cash Balance, December 31, 1939		$750.73	

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance, January 1, 1939:			
Principal—Investment (at Cost)	$6 625.00		
Income—Cash	693.90	$7 318.90	
Receipts			
Interest on Deposits and Investments		87.98	7 406.88
Disbursements			
Engrossing 1939 Lecture Certificates		3.25	
Honorarium, 1939 Lecturer		200.00	
Cost of 1939 Medal		35.00	
Miscellaneous		25.00	263.25
Balance, December 31, 1939:			
Principal—Investments (at Cost)		6 625.00	
Income—Cash		518.63	$7 143.63

COMMITTEE FUNDS

Committee A-1 on Steel	$756.08	
Committee A-5 on Corrosion of Iron and Steel	3 368.46	
Committee A-5 Subcommittee X on Embrittlement Investigation	138.34	
Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys	3 290.22	
Committee B-6 on Die-Cast Metals and Alloys	185.42	
Committee C-1 on Cement	978.59	
Committee C-1, Studies of Additions to Cement	107.62	
Committee C-1, Cement Reference Laboratory	800.31	
Committee C-9 on Concrete and Concrete Aggregates	186.72	
Committee C-9, Sanford E. Thompson Medal Fund	552.00	
Committee C-15 on Manufactured Masonry Units	310.36	
Committee C-18 on Natural Building Stones and Slate	171.76	
Committee D-1 on Paint, Varnish, Lacquer, and Related Products	109.62	
Committee D-2 on Petroleum Products and Lubricants	91.78	
Committee D-13 on Textile Materials	278.86	
Committee D-13, Testing Textile Finishes	2 593.53	
ASA Sectional Committee on Specifications for Cast-Iron Pipe and Special Castings	233.60	
ASA Sectional Committee on Classification of Coals	1 184.60	
Research Committee on Fatigue of Metals	170.39	
Total Committee Funds		$15 508.26
Accounted for as Follows:		
Cash Balance, December 31, 1939	$9 508.26	
Investments—at cost	6 000.00	$15 508.26

INVESTMENTS, DECEMBER 31, 1939

GENERAL FUNDS

Number of Shares or Par Value	*Bonds*	*Book Value*	*Market Value*[a]
$9 000	Baltimore & Ohio R. R. Refunding and General Mortgage Bonds, Series F, 5's, due 1996	$9 106.55	$2 610.00
4 000	New York, Chicago & St. Louis R. R. Refunding Mortgage Bonds, Series C, 4½'s, due 1978	2 805.61	2 300.00
3 000	Wheeling Steel Corp., First Mortgage Sinking Fund Bonds, Series A, 4½'s, due 1966	3 045.00	2 932.50
3 000	Republic Steel Corp., General Mortgage Bonds, Series B, 4½'s, due 1961	3 000.00	2 846.25
2 000	North Station Industrial Bldg., Inc., First Mortgage Sinking Fund Bonds, 5's, due 1962	1 850.00	1 200.00
3 000	Armour and Co., First Mortgage, 20-yr. Sinking Fund Bonds, Series C, 4's, due 1957	2 955.00	2 985.00
3 000	Pennsylvania State Water Corp., First Collateral Trust Bonds, 4¼'s, due 1966	2 850.00	3 105.00
3 000	Michigan Consolidated Gas Co., First Mortgage 4's, due 1963	2 925.00	3 045.00
3 000	Columbia Gas & Electric Co., Debentures, 5's, due 1961	3 038.00	3 127.50
500	Florida Power & Light Co., First Mortgage, 5's, due 1954	491.25	518.13
	Stock		
50	shares Great Northern Ry. $6.00 Preferred Stock, par value $100	1 774.83	1 337.50
50	shares General Electric Co., Common Stock, no par value	2 006.25	2 018.75
100	shares American Radiator & Standard Sanitary Corp., Common Stock	1 564.32	1 012.50
25	shares American Tobacco Co., Common Stock	2 110.12	2 125.00
25	shares Hazel Atlas Glass Co.	2 510.64	2 806.25
25	shares Sears, Roebuck & Co., Capital Stock	1 760.71	2 128.13
1	share Standard Oil Co. of New Jersey, Capital Stock	21.37	43.88
200	shares United Gas Improvement Co., Common Stock	2 578.00	2 900.00
20	shares West Penn Power Co., Preferred Stock, par value $100.	2 200.00	2 295.00
50	shares United States Gypsum Co.	3 819.27	4 162.50
100	shares S. S. Kresge Co., Common Stock	2 490.18	2 462.50
		$54 902.10	$47 951.39

A.S.T.M. RESEARCH FUND

	Bonds		
$1 500	Baltimore & Ohio R. R. Refunding and General Mortgage Bonds, Series F, 5's, due 1996	1 511.25	435.00
2 000	New York, Chicago & St. Louis R. R. Refunding Mortgage Bonds, Series C, 4½'s, due 1978	1 400.26	1 150.00
2 000	First Mortgage Real Estate Bonds, 1315–1317 Spruce St., Philadelphia, 6's, due 1945	2 000.00	1 200.00
3 000	North Station Industrial Bldg., Inc., First Mortgage Sinking Fund Bonds, 5's, due 1962	2 812.50	1 800.00
1 000	Baldwin Locomotive Works, First Mortgage, Sinking Fund Bonds 5's, due 1940	991.53	1 012.50
1 000	Allis Chalmers Mfg. Co., Convertible Debentures, 4's, due 1952.	1 035.28	1 105.00
1 000	Columbia Gas & Electric Co., Debentures, 5's, due 1961	1 012.65	1 042.50
1 500	Florida Power & Light Co., First Mortgage, 5's, due 1954	1 473.75	1 554.38
	Stock		
29	shares Consumers Power Co., $4.50 Preferred Stock, no par value	2 914.50	2 939.88
98	shares General Motors Corp., Common Stock, par value $10[b]	6 504.75	5 341.00
12	shares Consolidated Edison Company of New York, $5.00 Preferred Stock	1 234.39	1 293.00
		$22 890.86	$18 873.26

DUDLEY MEDAL AND MARBURG LECTURE FUND

Number of Shares or Par Value		*Book Value*	*Market Value*[a]
$500	Baltimore & Ohio R. R. Refunding and General Mortgage Bonds, Series C, 6's, due 1995	550.00	152.50
6 000	Baltimore & Ohio R. R. Refunding and General Mortgage Bonds, Series A, 5's, due 1995	6 075.00	1 740.00
		$6 625.00	$1 892.50

COMMITTEE FUNDS

$5 000	U. S. Treasury Bonds, $2\frac{3}{4}$'s, due 1947	5 000.00	5 456.25
1 000	U. S. Treasury Bonds, $2\frac{7}{8}$'s, due 1960	1 000.00	1 078.75
		$6 000.00	$6 535.00
		$90 417.96	$75 272.15

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[a] Market Values, except on First Mortgage Real Estate 1315-1317 Spruce St., 6's, 1945, on which there was no quotation, and North Station Industrial Bldg., Inc., First Mortgage Sinking Fund, 5's, 1962, on which a quotation was obtained from the brokers, were taken from the Wall Street Journal of December 30, 1939.

[b] The income from these shares is by agreement paid to an annuitant.

in 1939 and the $12,000 reserve continued to be carried as a liability for the balance of the cost of the complete Book of Standards. The sum of $3000 reported as a liability for Book of Standards Reserve Account for Sales for Future Delivery is explained by the fact that charges totaling approximately $9000 for orders of the complete Book of Standards were placed on the books in December, 1939, although delivery of these orders could be made only on Part III; accordingly it was thought best to carry one-third of this sum as a liability.

The auditors' report includes a comparison of the financial condition of the Society at the close of the last five years. The surplus in the general funds account, exclusive of special and designated funds, is $58,117.14, which compares with $57,332.84 a year ago. The principal of the Research Fund on December 31, 1939, was $24,282.68.

Investment of Society funds as of December 31, 1939, are given in the auditors' report, both book and market values being shown. In line with the policy of increasing the proportion of Society General Funds invested in dividend-paying common stock, various purchases of such stock were made during the year from surplus. Sales of certain of these stocks before the close of the year yielded a profit of $1350, and the stocks remaining in the Society's portfolio on December 31 are listed in the auditors' report. Uninvested principal in the A.S.T.M. Research Fund at the close of 1938 was invested in 12 shares of Consolidated Edison Co. of New York $5 preferred stock. Certain transfers of investments were made affecting both General and Research Funds: $2000 Saguenay Power Co. First Mortgage $4\frac{1}{4}$ per cent Bonds were replaced with $2000 Florida Power and Light Co. First Mortgage 5 per cent bonds; 40 shares of Northern States Power $5 preferred stock were replaced with $4000 Columbia Gas and Electric Co. 5 per cent debenture bonds; $4000 Montana Power Co. First Mortgage $3\frac{3}{4}$ per cent bonds were replaced with 40 shares of common stock of American Can Co., which were subsequently sold and the proceeds reinvested in another common stock.

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock, with the exception of the cost value, $941.47, of the copies still in stock of the 1931 Joint A.S.M.E.-

A.S.T.M. Symposium on Effect of Temperature on the Properties of Metals purchased by the Society from a joint account; this amount is included in the statement of assets. There appears in the statement of liabilities the sum of $1100.72, which represents net profit to December 31, 1939, on the 1938 Joint A.S.T.M.-A.S.M.E. Creep Data Volume likewise published jointly and handled through a separate account as a "nonoperating" item. An inventory of technical publications on June 1, 1940, may be summarized as follows:

	Number of Copies
Year Book	275
Proceedings	5 027
Index to Proceedings, 5 parts, total	1 477
1939 Book of A.S.T.M. Standards:	
Part I	3 195
Part II	2 341
Part III	2 154
Index to Standards and Tentative Standards	2 552
Selected Standards for Students in Engineering	4 317
Reprints of Standards and Tentative Standards (approximate)	55 000
Symposium on Effect of Temperature on the Properties of Metals	212
Creep Data Volume	302
Special Reprints (approximate)	30 000

1940 Finances.—The Society's operations for 1940 are based upon budgeted operating receipts of $169,500. Receipts from dues and entrance fees are estimated at $84,250; receipts from sales of publications at $72,500, of which about half is expected to be realized from the sales of the 1939 Book of Standards and 1940 Supplement thereto; miscellaneous receipts, including advertising, interest and dividends, are estimated at $12,750. Disbursements have been estimated at $160,000, leaving an estimated surplus of about $9500 to be applied if necessary to operations in the second and third years of the triennial period beginning with 1940. The budget provides fully for all the Society's normal operating expenses including all current publications, activities of standing and district committees and necessary expansion of staff, to which reference is made later. The Executive Committee reviews the budget of receipts and disbursements quarterly and makes any adjustment that may be necessary.

Payment of the costs of the 1939 Book of Standards, totalling $27,088.92, was completed in May, 1940, by the payment of $10,109.75 from the $12,000 Book of Standards Reserve, which together with the sum of $16,979.17 paid in 1939 (see auditors' report) makes up the total. This does not include the costs of resetting standing material in the double-column format. These costs have amounted to $13,919.25, which includes about $3000 for editorial services not covered in the usual budget of salaries; a total of $10,119.25 has been paid—$2128.77 in 1938, $3988.09 in 1939 (see auditors' report), $4002.39 in 1940—leaving only $3800 to be carried over for future payment in accordance with the arrangements made with Waverly Press, the Society's printers. A balance of $1890.25 remains in the Book of Standards Reserve for application to the costs of the 1942 edition.

Investments of general funds have been made so far this year in common stocks of J. C. Penney Co., Public Service Corp. of New Jersey, Atlantic Refining Co., and Mathieson Alkali Co., and in $2.50 preferred stock of General Telephone Co. Twenty shares of West Penn Power $4.50 preferred stock were sold and the proceeds invested in 25 shares of Colgate-Palmolive-Peet 4¼ per cent preferred stock. The sum of $6000 from 1940 receipts applied to the Book of Standards Reserve has been invested in shares of two Federal Savings and Loan Associations, currently paying 4 per cent—30 shares of value $3000 in each association. Deposits in these associations up to $5000 are guaranteed by the Federal Savings and Loan Insurance Corporation. These investments, of course, do not appear in the present auditors' report. They bring the total book value of Society investments as of May 31, 1940, in all funds, to $111,621.69; market value of these securities on the same date was $79,501.40. The Society's cash balance on May 31, 1940, was $64,765.42.

Expansion of Staff:

In its report a year ago the Executive Committee referred to some of the problems, administrative and financial, that are presented by the rapid growth of the standardization work of the Society and expansion of our activities into new industrial fields. An essential need is for an enlarged editorial staff capable of handling promptly and efficiently the editing of our technical specifications and tests, which are often of highly special nature requiring competent and experienced persons with suitable training. The Executive Committee has been able partly to meet this need by the appointment to the staff of Mr. M. D. Huber, a graduate in chemistry, and by augmenting the clerical staff when necessary to provide adequate help at periods of peak publication activity. This enlargement of the staff has been made possible by the added income available from sustaining memberships. These memberships, moreover, have enabled the Society to meet from current income the steadily growing expense of publication of standards and to plan ahead for future developments with greater assurance,—as for example, the further growth of the BULLETIN as a technical journal. Sales of the Book of Standards and its supplements are counted upon to provide further net income in support of publications, and while present indications are promising it will require a three-year period beginning with this year to determine the extent and relative certainty of that source of income.

Retirement Plan for Employees:

Effective May 1, 1940, the Executive Committee has increased the Society's contribution to the Retirement Plan for A.S.T.M. Employees by $25 per month, bringing the annual contribution to $1380 or about 2½ per cent of the salary roll. These contributions are applied to the purchase of retirement income policies in the Penn Mutual Life Insurance Co. of Philadelphia for the eighteen (of twenty-one) employees who are participating, each employee matching from his salary the Society's contribution towards his retirement policies. Nearly all these employees are contributing 2½ per cent or more of their salaries and the ratios between monthly retirement income (at age 65 for men and 60 for women) and salaries are in some cases approaching desirable percentages.

The sums of $500 and $250 were applied in 1939 from surplus towards retirement income for the Secretary-Treasurer and Assistant Secretary, respectively. In the present fiscal year (1940) the sum of $250 was similarly applied for the Assistant Secretary. In addition an Executive Retirement Reserve has been established for the purpose of providing funds towards requirements for retirement of members of the executive staff of the Society supplementary to retirement income policies provided under the Retirement Plan, to which Reserve there has been applied from surplus the sum of $1000 with the provision that income from this sum shall be added to the principal.

Headquarters:

The space in Society headquarters devoted to addressograph and duplicating operations, shipping and publication storage, heretofore on the fourth floor of the building, has been increased about 20 per cent in taking new rooms on the same floor (fifth) on which the main offices are located. Shelving space in new steel stacks for publication storage has been increased about 30 per cent and better arrangement of facilities has been effected. Annual rental has increased in amount commensurate with the increased space acquired, and the lease on the entire headquarters, now comprising about 4200 sq. ft., has been extended to December 31, 1942.

Relationship with Program for Industrial Preparedness:

The importance of materials standards in the Government's Industrial Preparedness Program has led the Executive Committee to discuss with appropriate officials of the Government the relationship of the Society to the plans for industrial mobilization in the event of emergency. The standardization and other facilities of the Society have been offered to the Planning Division, Office of the Assistant Secretary of War, and ways in which these facilities including our extensive committee organization might be utilized have been suggested.

These discussions have emphasized the importance in time of emergency of having A.S.T.M. specifications and Federal specifications as closely in harmony as to substance as possible, and plans are under development—for initial application to the metals field—to bring about where possible a closer and more direct contact between A.S.T.M. and Federal committees when specifications or their revisions are in the formative stage. Increased representation of various branches of the War Department upon the committees of the Society has been arranged, and in one important instance—that of copper and copper alloys, Committee B-5—has already resulted in effective cooperation in the development of specifications for materials of great importance in the production of munitions, which are being offered to the Society at this meeting. Committee D-11 has also been cooperating effectively with the Federal Specifications Committee on Rubber.

Cooperative Activities:

The Society's activities have always of necessity involved cooperative relations with many other organizations, which relations proceed along well-established lines. News items in the ASTM BULLETIN have told of various activities in this direction and these need not be repeated. Several happenings during the year, however, merit specific mention here.

The Society's contacts with the American Standards Association constitute one of its most important cooperative activities. During the past year the principal work has been more or less routine in nature, involving the submission of new and revised standards to the ASA for approval as American standard. The annual report of Committee E-10 on Standards presents this work in detail.

Upon nomination of the Society, Jerome Strauss, Vice-President, Vanadium Corporation of America, was elected to membership on the Board of Directors of the American Standards Association, succeeding Dr. G. W. Thompson who had resigned. President H. H. Morgan was re-appointed for a term of three years as one of the Society's representatives on the Standards Council. R. P. Miller has been reappointed as the Society's representative on the Building Code Correlating Committee.

The cooperative work with the Society of Automotive Engineers in the development of standards for automotive rubber, begun over a year ago and mentioned in last year's report, is assuming much importance and is being actively carried on by a joint committee of technical men from the rubber and automotive industries that is functioning as Technical Committee A of Committee D-11 on Rubber Products.

Interest in the work of the Inter-Society Color Council is seen in the appointment of three additional nonvoting delegates from the Society in the persons of C. E. Foss, A. G. Ashcroft and C. J. Huber. The Society's voting and in that sense official delegates are M. Rea Paul, A. W. Kenney and W. M. Scott. The Color Council is sponsoring a symposium on "The Use of Color in the Testing of Materials" at the Spring Meeting of the Society to be held next March in Washington.

During the year the Society has cooperated with the Analytical Division of the American Chemical Society in developing plans for a symposium on the subject "Tools of Analytical Chemistry" which is one of the features of the present meeting.

The Society was represented at the Eighth American Scientific Congress held in Washington, May 10 to 18, 1940, by Past-President T. G. Delbridge, who presented a paper before Section VI of the Congress on Physical and Chemical Sciences on the subject "Quality Measurements of Petroleum Products." At the suggestion of the Society W. D. Appel of the National Bureau of Standards presented a paper before the same Section describing developments in this country on specifications and tests for textiles.

The Joint Committee on Trolley Wire Specifications, which had existed for many years as a committee of the American Transit Association, Association of American Railroads and this Society, has by agreement among the constituent organizations been disbanded and its work turned over to the Society's Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors, to which representatives of the other two associations have been added.

Respectfully submitted on behalf of the Executive Committee,

H. H. MORGAN,
President.

C. L. WARWICK,
Secretary-Treasurer.

June, 1940.

REPORT OF COMMITTEE A-1

ON

STEEL

Prominent among the standardization activities of Committee A-1 on Steel during the past year has been the intensive work involving forgings, spring wire, pipe and tubing, and materials for high-temperature service including pipe, castings, and forgings. As discussed in this report under activities of subcommittees, a number of important recommendations have been developed involving both new specifications and revisions of existing tentative standards and standards, most of the latter being offered for immediate adoption.

Three series of meetings have been held during the year: the first in Atlantic City, N. J., on June 26 and 27, 1939; the second in Philadelphia, Pa., on January 8 and 9, 1940; and the third in Detroit, Mich., on March 4 and 5, 1940. Including the sessions of subcommittees and sections, the number of meetings held during the year was in excess of 50. Members have cooperated closely by attendance at these meetings and the committee's work in the development of purchase specifications has been notably advanced.

Since the 1939 annual meeting, 12 new members have been elected and 4 resignations accepted, the committee membership now totaling 229, of whom 121 are classified as nonproducers and 108 as producers; of the former, 93 are consumer and 28 general interest members.

Work by Sections.—While some of the subcommittees have organized permanent sections to handle particular phases of their work, and other subcommittees informally have appointed sections during recent years to develop recommendations or to investigate certain specific problems, there has been a notable increase in the use of this method of handling various problems confronting the committee. Except in its minutes the committee does not officially list the personnel of these sections but recognition of their work is noted from time to time in the committee reports. Sections are frequently constituted with equal representation from non-producers and producer interests and in all cases there is representation of active consumers. Too much credit cannot be given these various groups for their intensive work. Frequently, the extensive correspondence and the long hours spent by the members at meetings, are effective in coordinating different viewpoints and conserving the efforts of the larger groups constituting the subcommittees and Committee A-1 itself. Many of the subcommittees have a very extensive membership personnel in order to cover effectively the different types and uses of material, such as forgings, pipe and tubing, materials for high-temperature service, and the like. The use of the section method in handling work insures concentrated efforts of authorities on a specific subject.

Losses by Death.—During the year Committee A-1 has lost by death three members. The committee records its sincere appreciation of their generous services. E. E. Hughes, one of the founders of the Rail Steel Bar Association, was particularly concerned, until his gradual retirement from active business, with steel concrete reinforcement. C. W. Johns, Chesapeake and Ohio Railway Co., although not active in the committee over a long period of time, had been keenly interested in certain work. Special mention should be made of the sustained interest of R. S. MacPherran, Allis-Chalmers Manufacturing Co. A member of the committee since 1914, he had been active in the work of several subcommittees, including Subcommittee VI on Steel Forgings and Billets, Subcommittee VIII on Steel Castings, and in the work on bar steels. An outstanding metallurgical authority, Mr. MacPherran was a loyal supporter of the work of the Society, particularly, of its work involving iron and steel. His loss will be felt keenly by his associates in committee work. Committee A-1 expresses its deep appreciation of his interest.

Tentative Standards and Revisions Accepted During the Past Year.—Subsequent to the 1939 annual meeting, Committee A-1 presented to the Society through Committee E-10 on Standards the following recommendations, which were accepted[1] by Committee E-10 on August 24, 1939:

Tentative Specifications for:

Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 39 T),

Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 39 T),

Hard-Drawn Steel Spring Wire (A 227 – 39 T),

Steel Music Spring Wire (A 228 – 39 T),

Oil-Tempered Steel Spring Wire (A 229 – 39 T),

Carbon-Steel Valve Spring Quality Wire (A 230 – 39 T),

Chromium-Vanadium Steel Spring Wire (A 231 – 39 T), and

Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 – 39 T).

Revision of Tentative Specifications for:

Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures (A 193 – 39 T),

Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 39 T), and

Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 – 39 T).

Tentative Revision of Standard Specifications for:

Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 37), and

Carbon-Steel and Alloy-Steel Forgings (A 18 – 30), in the form of new Tentative Specifications for Carbon-Steel Forgings (A 18 – 39 T).

Withdrawal of Standard Specifications for:

Marine Boiler Steel Plates (A 114 – 33).

The new and revised tentative specifications and the tentative revisions of the standard specifications appear in the 1939 Book of A.S.T.M. Standards, Part I.

The committee also presented to the Society through Committee E-10, new Tentative Specifications for Iron and Steel Arc-Welding Electrodes (A 233 – 40 T) and a recommendation to withdraw the Tentative Specifications for Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods) (A 205 – 37 T). These recommendations were accepted[2] by Committee E-10 on May 1, 1940, and the new tentative specifications have been issued.[2a]

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee A-1 reported the following results of the letter ballot vote of a total of 176 ballots returned from a committee membership of 226: A 225 - 39 T, affirmative 93, negative 1, ballots marked "not voting" 82; A 226 - 39 T, affirmative 84, negative 0, ballots marked "not voting" 92; A 227 - 39 T, affirmative 82, negative 0, ballots marked "not voting" 94; A 228 - 39 T, affirmative 77, negative 2, ballots marked "not voting" 97; A 229 - 39 T, affirmative 80, negative 0, ballots marked "not voting" 96; A 230 - 39 T, affirmative 78, negative 0, ballots marked "not voting" 98; A 231 - 39 T, affirmative 78, negative 1, ballots marked "not voting" 97; A 232 - 39 T, affirmative 77, negative 1, ballots marked "not voting" 98; A 193 - 39 T, affirmative 88, negative 0, ballots marked "not voting" 88; A 206 - 39 T, affirmative 86, negative 0, ballots marked "not voting" 90; A 217 - 39 T, affirmative 101, negative 0, ballots marked "not voting" 75; A 178 - 37, affirmative 84, negative 0, ballots marked "not voting" 92; A 18 - 30, affirmative 115, negative 4, ballots marked "not voting" 57; A 114 - 33, affirmative 75, negative 1, ballots marked "not voting" 100.

The six new specifications for spring wire came about as a result of a request from a large consumer using spring wire that the Steel Committee consider developing standard requirements for this material. While Subcommittee IV, to which the problem was referred, realized the many problems in this field because of the wide variety of uses of spring wire and the different grades and classes of material, intensive study by one of its sections resulted in the conclusion to draft requirements not only for the so-called music spring material but for five other widely used types of wire.

Discussion of the new specifications for carbon-steel forgings (A 18) appears in a later portion of this report under activities of subcommittees.

Since there appeared to be justification for standardized requirements for electric-resistance-welded steel boiler and superheater tubes for high-pressure service, requirements were drafted by a special section of Subcommittee IX on Steel Tubing and Pipe.

Subcommittee XI on Boiler Steel has developed various specifications for so-called low alloy plates for use in the construction of boilers and other pressure vessels. A demand for specifications for manganese-vanadium steel plates resulted from rather widespread use of this material.

Changes in the specifications for alloy-steel bolting materials for high-temperature service (A 193) were for the purpose of facilitating the ordering of material and to indicate in the table of physical properties certain material which might be considered as of stock status. Also, specific heat treatments were incorporated. The changes in the seamless carbon-molybdenum alloy-steel pipe specifications (A 206) incorporate additional requirements on grain size. The changes in the requirements for electric-resistance-welded steel boiler tubes (A 178), when adopted, will bring the tables of weights and permissible variations in line with similar specifications using the decimal system instead of the old B.w.g. setup. They will also provide decreased tolerances and other modifications.

The withdrawal of the Standard Specifications for Marine Boiler Steel Plates (A 114 – 33) was recommended because the specifications were obsolete in many respects. Their use was limited due to certain regulations of various naval and marine bureaus.

The specifications for arc-welding electrodes (A 233 – 40 T), developed jointly through the Filler Metal Specifications Committee of the American Welding Society, J. H. Deppeler, chairman, and the Steel Committee's Subcommittee XXI on Steel for Welding, N. L. Mochel, chairman, were in course of preparation for many months and the subject of much correspondence and numerous meetings. They resulted from the growing conviction that the existing Specifications for Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods) (A 205 – 37 T) were obsolete in many respects. It was recommended that concurrent with the approval of the new specifications, the older ones be withdrawn, even though the former do not cover gas-welding rods. It is the intent of the respective committees to develop additional specifications for gas-welding rods and also to cover materials suitable for welding the higher alloyed steels. The new specifications cover electrodes for the welding of carbon and low-alloy steels of weldable quality. They are classified on the basis of usability and ultimate tensile strength of all-weld-metal specimens in the stress-relieved condition. There are some 14 different electrode classifications.

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[2] In submitting these recommendations to Committee E-10 on Standards, Committee A-1 reported results of the letter ballot vote as follows: Of a total membership of 226, 156 members returned their ballots, of whom 106 voted affirmatively, 4 negatively, and 46 members marked their ballots "not voting."

[2a] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 193.

For convenience, the recommendations of the committee affecting its standard and tentative specifications are listed below in summarized form with an analysis of the letter ballot vote given in Table I. Certain of these recommendations are referred to further in the discussion of subcommittee activities.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following specifications be accepted for publication as tentative:

Tentative Specifications for:[3]

Carbon-Steel Forgings for General Industrial Use,

Carbon-Steel Forgings for Locomotives and Cars,

Alloy-Steel Forgings for General Industrial Use,

Alloy-Steel Forgings for Locomotives and Cars, and

Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following tentative specifications be revised, as indicated in the Appendix, and continued as tentative:

Tentative Specifications for:

Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 – 39 T),

Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 38 T),

Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures (A 193 – 39 T),

Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 – 39 T),

Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 – 39 T),

Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F (A 158 – 38 T), and

Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 39 T).

III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption, revisions in the following standards, and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society for adoption as standard:

Standard Specifications for:

Steel **Plates** of Structural Quality for Forge Welding (A 78 – 39),

Boiler Rivet Steel and Rivets (A 31 – 39),

Welded and Seamless Steel Pipe (A 53 – 36),

Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 36),

Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 37),

Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 – 38),

Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 39),

Seamless Low-Carbon Steel Still Tubes for Refinery Service (A 161 – 37),

Seamless Alloy-Steel (4 to 6 per cent Chromium) Still Tubes for Refinery Service (A 188 – 38),

Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 39),

Commercial Quality Hot-Rolled Bar Steels (A 107 – 39),

Forged or Rolled Steel Pipe Flanges for High-Temperature Service (A 105 – 39),

Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for Service at Temperatures from 750 to 1100 F. (A 182 – 39),

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[3] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, pp. 204, 210, 217, 223, 269, respectively.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Spec. for Carbon-Steel Forgings for General Industrial Use	106	1	57
Spec. for Carbon-Steel Forgings for Locomotives and Cars	90	0	74
Spec. for Alloy-Steel Forgings for General Industrial Use	100	0	63
Spec. for Alloy-Steel Forgings for Locomotives and Cars	87	0	77
Spec. for Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings	66	4	94
II. Revisions of Tentative Standards			
Spec. for Heat-Treated Steel Track Bolts and Nuts (A 183 - 39 T)	57	1	88
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 38 T)	82	0	82
Spec. for Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures (A 193 - 39 T)	85	0	62
Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 - 39 T)	83	1	80
Spec. for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 39 T)	83	1	80
Spec. for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 - 38 T)	78	0	90
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 39 T)	72	2	90
III. Revisions of Standards, Immediate Adoption			
Spec. for Steel Plates of Structural Quality for Forge Welding (A 78 - 39)	87	4	55
Spec. for Boiler Rivet Steel and Rivets (A 31 - 39)	73	0	91
Spec. for Welded and Seamless Steel Pipe (A 53 - 36; ASA No. B36.1-1936)	84	0	63
Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 36; ASA No. G8.7-1937)	81	1	82
Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 37)	80	0	84
Spec. for Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 - 38)	81	0	83
Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 39)	80	0	84
Spec. for Low-Carbon Steel Still Tubes for Refinery Service (A 161 - 37)	80	1	65
Spec. for Seamless Alloy-Steel (4 to 6 per cent Chromium) Still Tubes for Refinery Service (A 188 - 38)	76	1	87
Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 39)	76	1	87
Spec. for Commercial Quality Hot-Rolled Bar Steels (A 107 - 39)	83	0	63
Spec. for Steel Pipe Flanges for High-Temperature Service (A 105 - 39)	74	0	73
Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for Temperatures from 750 to 1100 F. (A 182 - 39)	78	0	68
Spec. for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 39; ASA No. B36.3-1939)	75	2	69
Spec. for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 - 36; ASA No. G17.1-1936)	83	1	80
Spec. for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 - 39)	84	0	80
Spec. for Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service to 1100 F. (A 194 - 39)	87	0	60
IV. Tentative Revisions of Standards			
Spec. for Structural Steel for Locomotives and Cars (A 113 - 39)	82	0	64
Spec. for Steel Castings for Miscellaneous Industrial Uses (A 27 - 39)	93	0	54
Spec. for Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 - 36)	93	0	54
Spec. for Alloy-Steel Castings for Structural Purposes (A 148 - 36)	93	0	54
Spec. for Forged or Rolled Pipe Flanges for General Service (A 181 - 37)	74	0	73
Spec. for Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 - 36; ASA No. B36.11-1939)	74	0	73
V. Adoption of Tentative Standard as Standard			
Spec. for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 38 T), as revised	82	0	65
Spec. for Seamless Boiler Tubes for High-Pressure Service (A 192 - 38 T)	79	0	68
Spec. for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 - 38 T), as revised	82	0	65
Spec. for Spiral-Welded Steel or Iron Pipe (A 211 - 38 T), as revised	73	1	90
Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 39 T)	79	0	68
Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 39 T), as revised	77	0	70
Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 39 T), as revised	76	0	71
VI. Adoption as Standard of Tentative Revisions of Standards			
Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 37)	80	0	67
Spec. for Boiler Rivet Steel and Rivets (A 31 - 39)	73	0	91
VII. Withdrawal of Standards and Tentative Standards			
Spec. for Carbon-Steel and Alloy-Steel Forgings (A 18 - 30)	90	0	74
Spec. for Normalized-and-Tempered Alloy-Steel Forgings (A 63 - 39)	90	0	74
Spec. for Normalized Alloy-Steel Forgings for Locomotives (A 133 - 33 T)	90	0	74
Spec. for Carbon-Steel Forgings (A 18 - 39 T)	90	0	74

Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 39),

Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 - 36),

Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 - 39), and

Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service to 1100 F. (A 194 - 39).

IV. TENTATIVE REVISIONS OF STANDARDS

The committee recommends for publication as tentative, revisions as given in the Appendix, of the following standards:

Standard Specifications for:

Structural Steel for Locomotives and Cars (A 113 - 39),

Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 - 39),

Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 - 36),

Alloy-Steel Castings for Structural Purposes (A 148 - 36),

Forged or Rolled Steel Pipe Flanges for General Service (A 181 - 37), and

Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 - 36).

V. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the following tentative specifications be referred to letter ballot of the Society for adoption as standard, with revisions as given in detail in the Appendix:

Tentative Specifications for:

Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 38 T),

Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 38 T),

Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 - 38 T),

Spiral-Welded Steel or Iron Pipe (A 211 - 38 T),

Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 39 T),

Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 39 T),

Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 39 T),

VI. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

The committee recommends that the revisions published as tentative last year[4] in the Standard Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 37) and for Boiler Rivet Steel and Rivets (A 31 - 39) be approved for reference to letter ballot of the Society for adoption as standard.

VII. WITHDRAWAL OF STANDARDS AND TENTATIVE STANDARDS

The following specifications[5] are recommended for withdrawal:

Standard Specifications for:

Carbon-Steel and Alloy-Steel Forgings (A 18 - 30), and

Normalized Quenched-and-Tempered Alloy-Steel Forgings (A 63 - 39).

Tentative Specifications for:

Normalized and Tempered Alloy-Steel Forgings for Locomotives (A 133 - 33 T), and

Carbon-Steel Forgings (A 18 - 39 T).

The recommendations appearing in this report have been referred to letter ballot of the committee which consists of 229 members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Certain features of the committee's work are discussed below, grouped in the order of the respective subcommittees responsible.

Special Subcommittee on Weldability (N. L. Mochel, chairman).—Because several of the Steel Committee's standing subcommittees are concerned with questions involving the weldability of steel, in particular, the development of reliable and reasonably quick tests to determine this quality, it was decided to organize a special subcommittee to consider various problems in this field.

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[4] 1939 Book of A.S.T.M. Standards, Part I, pp. 1271 and 1273.

[5] *Ibid.*, pp. 184, 192, 821, 816.

An all-day organization meeting of the subcommittee was held in Philadelphia, Pa., on October 3, 1939, with the chairman of Committee A-1 presiding. There were representatives present from four of the subcommittees chiefly concerned: namely, Subcommittees II on Structural Steel for Bridges, Buildings, and Rolling Stock, VIII on Steel Castings, XI on Boiler Steel, and XXII on Valves, Fittings, Piping and Flanges for High-Temperature Service.

After extended discussion of many involved factors, Mr. Mochel, who was chosen by the members to act as chairman of the special subcommittee, was asked to appoint a section to prepare recommended practices for weld testing, including all such tests as may be useful to all subcommittees, to establish the suitability and uniformity of steels for welding.

A meeting of this section, consisting, in addition to the chairman, of G. F. Jenks, A. B. Kinzel, H. J. French, L. P. McAllister, and F. H. Frankland, was held during the January meetings of Committee A-1 in Philadelphia. Consideration of two general approaches, namely, the "data-chart" approach, which would involve a very comprehensive data sheet with all known information plotted, and the "application" or "acceptance test" approach, resulted in general agreement that the latter was preferable from the standpoint of the Steel Committee's problems. This would involve the development of what might be termed a "recommended practice" in which would be described the several testing methods or practices that are believed desirable for the testing of material intended for welding. From this practice, a subcommittee could then draw as many tests as would be desirable for its particular materials and assign necessary values for each.

The section is giving further consideration to this question. Meanwhile, it is developing further information and has requested that members of Committee A-1 who are concerned and have pertinent data should submit this to the chairman of the special subcommittee.

Subcommittee I on Steel Rails and Accessories (E. F. Kenney, chairman).—The revision in Section 9 on strip test of the Tentative Specifications for Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 – 39 T), as given in the Appendix, is recommended to overcome confusion which has occurred in applying this test. It is required that threads shall not strip at a load calculated from the specified minimum yield point and the area at the root of the bolt thread. Confusion has resulted from the general unfamiliarity with theoretical dimensions of threads. The table recommended for inclusion and the explanatory material simplify the necessary calculations.

These specifications require that the steel for the nuts is to conform to the resulfurized nut stock grades Nos. 26 and 27 of the Standard Specifications for Commercial Quality Hot-Rolled Bar Steels (A 107 – 39). Because of a change recommended by Subcommittee XV on Commercial Bar Steels in the latter specifications, involving the removal of the minimum limit for carbon, Subcommittee I recommends the change as shown in the Appendix. It is desirable for railroad track work that a minimum carbon content be required.

Subcommittee II on Structural Steel for Bridges, Buildings, and Rolling Stock (F. H. Frankland, chairman).—Since one of the changes proposed (see Appendix) in the Standard Specifications for Steel Plates of Structural Quality for Forge Welding (A 78 – 39) is of an urgent nature; namely, that the sulfur limit be changed, it is recommended that the other modifications involving number of tests also be adopted immediately, and that the use of the material for fusion welding be clarified by editorial changes. The change in chemistry will bring the requirements in line with current commercial classifications. The changes in the section on number of tests will bring these requirements in line with similar structural steel specifications. A similar change in the number of tests required in the Standard Specifications for Structural Steel for Locomotives and Cars (A 113 – 39) is recommended as a tentative revision, with the probability that it will be adopted as standard when the next Book of Standards is issued.

Subcommittee VI on Steel Forgings and Billets (W. M. Barr, chairman).—Following extended discussion at its meeting in June, 1939, the subcommittee recommended that the proposed standard published in the preprinted Report of Committee A-1 covering carbon-steel forgings be withdrawn because of a number of objections, particularly to the physical properties of the various classes of forgings. The subcommittee immediately developed changes in the specifications and the Society, through Committee E-10 on Standards on August 24, 1939, approved the revised Specifications for Carbon-Steel Forgings (A 18 – 39 T) as tentative; at the same time the existing Standard Specifications for Carbon-Steel and Alloy-Steel Forgings (A 18 – 30) were continued. Realization that some of the confusion in connection with standardized purchase requirements for forging arose from the fact that all types were covered in the existing specifications (that is, large and small, and those for railroads as well as those for general purposes) convinced the subcommittee that the logical procedure was to issue four specifications divided as to carbon steels and alloy steels, and also as to the application involved, whether for locomotives, cars and related uses, or for general industrial purposes, in which latter field larger forgings are frequently used.

Four sections were appointed, headed by P. E. McKinney, E. J. Edwards, J. H. Higgins, and A. O. Schaefer. With the close cooperation of other members of Subcommittee VI serving on one or more sections, the four tentative specifications listed earlier in this report have resulted. It is the intent of the subcommittee that with the approval of these as tentative all of the existing forging specifications will be withdrawn, except the Specifications for Carbon-Steel and Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 17 – 29), and for Carbon-Steel Axles for Cars and Tenders (A 21 – 36).

With some minor changes and the inclusion of another grade in the carbon-steel forgings for general industrial use, this grade having been suggested by those concerned with the structural fields—in particular, bridges—involving a minimum ultimate tensile strength of 66,000 psi. and a 33,000 psi. minimum yield point, the subcommittee believes the question of satisfactory purchase specifications for both carbon steel and alloy steel forgings has been distinctly clarified.

Subcommittee IX on Steel Tubing and Pipe (T. G. Stitt, chairman).—Of outstanding interest among the numerous recommendations of this subcommittee are the more rationalized standardized procedures for the hardness testing of boiler and superheater tubes and heat-exchanger and condenser tubes. The proposals, as detailed in the Appendix, whereby the Brinell hardness test will be made on the outside of tubes 0.200 in. and over in wall thickness, and the Rockwell test on the inside of a specimen cut from tubes less than 0.200 in. and down to 0.065 in., inclusive, eliminate certain inconsistencies and are helpful to the producer in standardizing procedures as well as providing other clarifications. With these changes in nine of the tube specifications are recommended modifications of the hardness number requirements of certain types of tubular materials, which bring these more nearly in line with possible commercial attainments. The recommendations involving hardness testing procedures and values were developed in a section headed by R. L. Wilson.

Another accomplishment of the subcommittee during the year was the preparation of an article[6] on hydrostatic test pressures for pipe, including proposed standard specification requirements. Based upon an extensive study by a section under the chairmanship of H. R. Redington, there were prepared three tables of test pressures, one a complete and detailed table listing bursting pressures for each size of pipe and wall thickness and including the values for test pressures recommended by this section, based on a study of the ability of the various producing mills to test with present equipment. This table was made comprehensive so that modifications of it can be applied to various specifications covering pipe sizes and walls and in the schedule number classification. The second table provides proposed test pressures for certain grades grouped in five size ranges. This proposal will greatly reduce the number of test pressures and make for economy and simplification in testing various sizes and weights. A modification of it, as given in the Appendix, is recommended for inclusion in the Specifications for Welded and Seamless Steel Pipe (A 53 – 36), and for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 36). A third table with one test pressure for all grades for each group of sizes under a given schedule number has been prepared for consideration in the revision of specifications for materials for use at high temperatures.

While the section in charge, which is comprised of leading producers and consumers of pipe and representatives from both Subcommittees IX and XXII, proposes that the maximum pressure be listed at 2800 psi. based on present average mill testing pump capacities, a revision can be set up, as is being done with certain materials for high-temperature service providing that by mutual agreement of producers and consumers and where the pump capacity is available the pipe will be tested to one and one-half times the working pressure.

The report of this section should make it possible eventually to remove the great many inconsistent requirements in the hydrostatic testing of pipe—a step desirable from the standpoint of standardized requirements and simplification.

To clarify requirements of the flattening test and remove a source of conflicting opinion, there will be added to all specifications where the flattening test is required a statement that: Superficial ruptures as a result of minor surface imperfections shall not be cause for rejection. This change will be incorporated in all specifications being revised, and in cases where specifications are not being changed but are being reprinted. While of a quasi-editorial nature, the committee has approved the recommendation by letter ballot and calls attention to the change by this reference.

Since the normal rotary straightening operation has not been entirely sufficient to remove scale from certain alloy-steel still and boiler tubes so that they are suitable for inspection, changes, as given in the Appendix, are proposed in the finishing clause of certain specifications.

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[6] ASTM BULLETIN, May, 1940, No. 104, pp. 25–30.

The change provides that pickling or shot-blasting can be used to remove scale and that in special manufacturing processes requiring this procedure it is to be done at the purchaser's expense.

With so many different kinds of pipe and tubing used in many different industries, and with widely different requirements, the subcommittee's work involves an extremely active field with new developments constantly arising, particularly in the field of alloy steels where new compositions are constantly coming into commercial importance, and others are losing favor. Periodic modifications of chemical requirements of existing grades are necessary and the addition of new alloys is frequently necessary. Several such changes are being recommended in various pipe and tubing specifications, as detailed in the Appendix.

Another project completed during the year is a detailed editorial study of seven specifications covering boiler and superheater tubes, to eliminate certain inconsistencies and to bring them as closely as possible into the same editorial style. Most of these changes will be incorporated this year because of other substance revisions in the specifications. Once a group of specificacations are in close conformity editorially, the revision or development of new standards can be carried out with a minimum of editorial study.

The committee will propose at this annual meeting the adoption as standard of existing tentative revisions in the Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 37) and also the adoption as standard of six existing tentative specifications covering various types of carbon and alloy steel boiler and superheater tubes.

A study of tolerances and finish requirements for upset ends of still tubes has resulted in rather extensive changes in portions of the specifications covering still tubes (A 161, A 188, and A 200).

The development of specification requirements for alloy-steel boiler tubes, carbon-molybdenum steel boiler tubes, and alloy-steel heat-exchanger tubes, all fabricated by welding processes, continues.

Subcommittee XI on Boiler Steel (E. J. Edwards, chairman).—The immediate adoption of the revisions of the Standard Specifications for Boiler Rivet Steel and Rivets (A 31 – 39), providing requirements for bending properties, as given in the Appendix, and the adoption of the tentative revisions published last year, as mentioned earlier in this report, providing chemical requirements and tensile properties, all of these changes applying to the proposed new grade of high-strength boiler-rivet steel, will provide complete requirements. The specifications as revised will cover the existing grade with a tensile strength of 45,000 to 55,000 psi. and the newer grade with a tensile strength of 58,000 to 68,000 psi.

Subcommittee XIII on Methods of Physical Tests (L. H. Fry, chairman). —In cooperation with manufacturers of testing machines, a section of this subcommittee has had tests under way on the effect of speed on yield point values and the correlation of hydraulic and screw driven testing machines as related thereto. The results of the work are expected to be made available to the subcommittee shortly.

During the year, the chairman has commented on various specifications and other matters involving the mechanics and terminology of testing, and has prepared a paper which is to be presented at this annual meeting on "Speed in Tension Testing and Its Effect on Yield

Point Values."[7] This incorporates data developed by Subcommittee XIII and some made available recently from other sources.

Subcommittee XV on Commercial Bar Steels (J. J. Shuman, chairman).—Since the major tonnage of bar steel used for the manufacture of nut stock appears to be in low-carbon grades arranged with varying compositions of carbon-manganese ratios so as to be suitable for manufacturing nuts by cold punching, it was felt desirable to recommend the revision, as given in the Appendix, involving the chemical composition of the open-hearth resulfurized nut grade 27 in the Standard Specifications for Commercial Quality Hot-Rolled Bar Steels (A 107 – 39).

Subcommittee XXII on Valves, Fittings, Piping, and Flanges for High-Temperature Service (A. E. White, chairman).—The preparation of the new tentative specifications for factory-made carbon steel and carbon molybdenum steel welding fittings for pressure piping, referred to earlier in this report, is related directly to the work of Sectional Committee B16 on Pipe, Flanges, and Fittings, particularly, the work on welding fittings. The sectional committee's dimensional and related standards will embody reference to the new material specifications, as developed by the Section on Welding Fittings headed by J. J. Kanter.

To make the marking clauses of the four specifications covering alloy steel and carbon steel castings for services at elevated temperatures (A 95, A 157, A 216, and A 217) consistent and in line with the system of marking established by the Manufacturers Standardization Society of the Valve and Fittings Industry, changes, as detailed in the Appendix, are recommended. The other changes in the Standard Specifications for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 – 39) are necessitated by a division of the 4 to 6 per cent chromium-molybdenum grade, C 5, into high- and low-silicon grades, which brings these compositions in line with recognized commercial practice and provides a setup much the same as in the alloy steel pipe specifications (A 158).

Increasing experience with the production and use of pipe covered by the Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 39 T) has indicated quite a number of changes which are being proposed, as given in the Appendix. Changes in the other pipe specifications in the charge of this subcommittee resulted from editorial study and are intended to clarify sections which have been misinterpreted, particularly the supplementary requirements. Changes in the hydrostatic test pressures are proposed as a result of extensive study by a special section, as mentioned under Subcommittee IX on Steel Pipe and Tubing.

As might be expected in a field of engineering, where applications of materials are quite special, with unusual problems entailed because of high operating temperatures, changes in composition of the material are not infrequent. These changes result in numerous recommendations on the part of the subcommittee to keep its standards in line with current trends.

The election of officers for the ensuing term of two years resulted in the selection of the following:

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[7] See p. 625.

Chairman, N. L. Mochel.
Vice-Chairman, E. F. Kenney.
Vice-Chairman, H. B. Oatley.
Secretary, H. P. Bigler.

This report has been submitted to letter ballot of the committee which consists of 229 members; 154 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

H. P. BIGLER,
Secretary.

R. J. PAINTER,
Staff Assistant.

APPENDIX

PROPOSED REVISIONS IN STANDARDS FOR STEEL

In this Appendix are given proposed revisions in specifications covering steel and steel products as referred to earlier in this report. These standards and tentative standards appear in their present form in the 1939 Book of A.S.T.M. Standards, Part I.

Revisions of Tentative Standards

Tentative Specifications for Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 - 39 T):

Section 3.—Change Paragraph (*b*) to read as follows by the addition of the italicized words:

(*b*) The steel for the nuts shall conform to the resulfurized nut stock grades Nos. 26 and 27 of the Standard Specifications for Commercial Quality Hot-Rolled Bar Steels (A.S.T.M. Designation: A 107) of the American Society for Testing Materials, *except that the minimum carbon percentage of Grade No. 27 shall be 0.15.*

Section 9.—Add a Paragraph (*b*) to this section to read as follows, relettering the present section as Paragraph (*a*):

(*b*) The actual loads in pounds applied to various standard sizes of bolts and grades of steel in accordance with the requirements in Paragraph (*a*) are prescribed in Table I.

TABLE I.

Note.—For the calculation of bolts of other diameters and threads per inch than those listed, the depth of the thread can be calculated as 0.649519 divided by the number of threads per inch.

Nominal Bolt Size, in. (Major Diameter)	Number of American National Standard Threads per Inch	Minor Diameter[a] (Diameter at Root of Threads)	Minimum Requirements of Strip Test, in Pounds		
			Carbon Steel		Alloy Steel
			Grade 1, Yield Point 70 000 psi.	Grade 2, Yield Point 80 000 psi.	Yield Point 85 000 psi.
¾	10	0.6201	21 140	24 160	25 670
⅞	9	0.7307	29 350	33 540	35 640
1	8	0.8376	38 570	44 080	46 840
1⅛	7	0.9394	48 520	55 450	58 910
1¼	7	1.0644	62 290	71 180	75 630

[a] Basic minor diameters are taken from Table No. 3, Coarse-Thread Series of the American Standard for Screw Threads (ASA No.: B1.1-1935).

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 38 T):

Section 4 (*a*).—In the tabulation of chemical requirements change the carbon content of grade T1 from "0.10 to 0.20 per cent" to read "0.08 to 0.20 per cent."

Section 7.—Make the same change in this section as recommended in Section 6 of Specifications A 192 - 38 T,[1] designating this new paragraph as (*c*).

Section 9.—Change from its present form to read the same as recommended in Section 8 (*a*), (*b*), (*d*), and (*e*) of Specifications A 192 - 38 T,[1] adding a sentence to Paragraph (*a*) to read as follows:

Hardness tests shall not be required on tubes less than 0.065 in. in wall thickness.

---

[1] See p. 83.

and also changing the last sentence of Paragraph (*b*) to read as follows:

> Except as specified in Section 11 (*c*), Rockwell hardness tests shall be made on the inside of a specimen cut from the tube.

Incorporate the present hardness test values in a new Paragraph (*c*) to read as follows:

> (*c*) The tubes shall have a hardness number not to exceed the following:

	Brinell Hardness Number (Tubes 0 200 in. and over in wall thickness)	Rockwell Hardness Number (Tubes less than 0.200 in. in wall thickness)
Grade T1.........	137	B77
Grade T1a........	143	B79

Section 12 (e).—Make the same change in this section as recommended in Section 11 (*b*) of Specifications A 192 - 38 T.[1]

Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures (A 193 - 39 T):

Table I.—Change the molybdenum content for grade B5 from "0.45 to 0.65 per cent" to read "0.40 to 0.60 per cent"; and change the silicon content for grades B7 and B7a from "0.15 to 0.25 per cent" to read "0.15 to 0.30 per cent."

Tentative Specifications for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 - 39 T):

Section 16.—Change from its present form to read as follows:

> 16. Pressure containing castings, made in accordance with either of the grades of steel covered by these specifications, shall be marked for material identification with the A.S.T.M. symbols for the grade of steel, WCA and WCB, respectively; and the melt number or melt identfiication. Marking shall be in such position as not to injure the usefulness of the castings.
>
> NOTE.—These marking requirements are taken from the Standard Marking System for Valves, Fittings, Flanges, and Unions (No. SP-25-1936) of the Manufacturers Standardization Society of the Valve and Fittings Industry.

Tentative Specifications for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 39 T):

Section 17.—Change from its present form to read as follows:

> 17. Pressure containing castings, made in accordance with any of the grades of steel covered by these specifications, shall be marked for material identification with the A.S.T.M. symbols for the grade of steel, WC1, WC1A, WC2, and WC4, respectively; and the melt number or melt identification. Marking shall be in such position as not to injure the usefulness of the castings.
>
> NOTE.—These marking requirements are taken from the Standard Marking System for Valves, Fittings, Flanges, and Unions (No. SP-25-1936) of the Manufacturers Standardization Society of the Valve and Fittings Industry.

Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 - 38 T):

Table I.—Change the chemical requirements for Grades P8a and P8b to read as prescribed in the accompanying Table II by the addition of the italicized figures and the omission of those in brackets.

Section 8 (a).—Under the tabulation of tensile properties, change the footnote to read as follows by the addition of the italicized figures and the omission of those in brackets:

> [a] For transverse tension tests the material shall conform to these minimum tensile requirements except that the elongation in 2 in. shall be [25] *20* per cent for the ferritic steels and 40 per cent for the austenitic steels.

Section 11.—Change Paragraph (*b*) to read as follows by the addition of the italicized words and the omission of those in brackets:

(*b*) The maximum hydrostatic test pressure shall not exceed 2500 psi. for *nominal* sizes 3 in. and under [in outside diameter, 4500 psi. for sizes over 3 in. to but not including $8\frac{5}{8}$ in. in outside diameter, or 3000 psi. for all sizes $8\frac{5}{8}$ in. and over in outside diameter] *or 2800 psi. for all nominal sizes over 3 in.* The hydrostatic pressure shall be maintained for not less than 5 sec.

TABLE II.—CHEMICAL REQUIREMENTS.

Class	Austenitic Steels	
Identification Symbol	P 8a	P 8b
Grade	18 Chromium-8 Nickel	18 Chromium 10 Nickel Stabilized with Titanium or Columbium
Carbon, max., per cent	0.07	0.08
Manganese, per cent	[0.20 to 0.70] *2.00 max.*	[0.70 max.] *3.00 max.*
Phosphorus, max., per cent	0.03	0.03
Sulfur, max., per cent	0.03	0.03
Silicon, per cent	0.75 max.	[0.50 max.] *0.75 max.*
Nickel, per cent	[8.00 to 10.00] *8.00 to 11.00*	[9.00 to 12.00] *9.00 to 13.00*
Chromium, per cent	17.00 to 20.00	[17.00 to 19.00] *17.00 to 20.00*
Molybdenum, per cent	......	......
Tungsten, per cent	......	......
Titanium, per cent	......	0.35 min.
or Columbium, per cent	......	[8 times carbon content, min.] *0.70 min.*

At the end of this section, add a note to read as follows:

NOTE.—When requested by the purchaser and so stated in the order, pipe in sizes 14 in. in nominal diameter and smaller shall be tested to one and one-half times the specified working pressure (where one and one-half times the specified working pressure exceeds 2800 psi.), provided the fiber stress corresponding to those test pressures does not exceed one half the minimum specified yield point of the material, as determined by the above formula.

Section 12 (*c*).—Change the fifth sentence of this section to read as follows by the addition of the italicized word and the omission of the word in brackets:

Specimens shall be flattened [hot] *cold* and heat treated in the same manner as the pipe, and shall be parallel between gage marks.

Section 14.—Add a footnote reference after the word "lot" to read as follows:

A lot shall consist of all the pipe bought by a purchaser of the same size and wall thickness from any one melt.

Section 19.—Add a new second sentence to Paragraph (*a*) to read as follows:

At the discretion of the inspector representing the purchaser, finished pipe shall be subject to rejection if surface defects acceptable under Paragraph (*c*) are not scattered, but appear over a large area in excess of what is considered "workmanlike finish."

Add a new first sentence to Paragraph (*b*) to read as follows:

All defects shall be explored for depth.

Delete Paragraph (*c*), and substitute the following:

(*c*) *Machining or Grinding Defects.*—Surface defects shall be classified and treated as follows:

(*1*) U-bottom mechanical marks and abrasions[a] excepting pits, tears, scabs, slivers, etc., shall be acceptable without grinding or machining, if not deeper than 12.5 per cent of the nominal wall thickness nor in any case deeper than $\frac{1}{16}$ in. All V-bottom marks and abrasions and U-bottom marks and abrasions deeper than $\frac{1}{16}$ in. but not deeper than 12.5 per cent of the nominal wall thickness shall be removed by grinding or machining to sound metal. Where the width of any mark or abrasion is less than the depth it shall be classified as V-bottomed.

(*2*) Pipe showing inside or outside surface checks (fish scale) $\frac{1}{64}$ in. or less in depth need not have these defects removed. Such defects over $\frac{1}{64}$ in. but not more than $\frac{1}{32}$ in. in depth shall be removed by machining or grinding. Pipe on which these defects are more than $\frac{1}{32}$ in. in depth shall be rejected, unless the manufacturer can demonstrate to the purchaser that the defects are not injurious as defined in Paragraph (*b*).

---

[a] Marks and abrasions are defined as cable marks, dinges, guide marks, roll marks, ball scratches, scores, etc.

(*3*) Pipe showing scabs, seams, laps, tears, or slivers not deeper than 5 per cent of the nominal wall thickness need not have these defects removed. If deeper than 5 per cent such defects shall be removed by machining or grinding providing they are not injurious as defined in Paragraph (*b*).

(*4*) Pits which do not fall under the classification of injurious defects as defined in Paragraph (*b*) need not be removed.

(*5*) When grinding or machining is permitted, the outside diameter at the point of grinding or machining may be reduced by the amount so removed. Should it be impracticable to secure a direct measurement, the wall thickness at the point of grinding, or at a defect not required to be removed, shall be determined by deducting the amount removed in grinding, or the depth of the defect, from the minimum measured wall thickness at the ends of the pipe, and the remainder shall not be less than 87.5 per cent of the nominal wall thickness.

(*6*) Machining or grinding shall follow the inspection at the mill of the pipe as rolled and shall be followed by supplementary visual inspection.

Section 21 (*a*).—At the end of this section add a sentence to read as follows:

When specified in the order, the manufacturer shall notify the purchaser in time so that he may have his inspector present to witness any part of the manufacture or tests that may be desired.

Supplementary Requirements. — Change Section S1 to read as follows by the addition of the italicized word:

S1. These requirements shall not be considered unless specified in the order, in which event the *following* supplementary tests shall be made at the mill, unless otherwise agreed upon, at the purchaser's expense and witnessed by his inspector before shipment of the material.

Change Section S3 to read as follows by the addition of the italicized sentence:

S3. Transverse tension tests may be made on specimens from both ends of each length of pipe. *This requirement applies only to pipe 8 in. and over in nominal diameter.* If the specimen from either end of any length fails to conform to the physical properties specified in Section 8, that length shall be rejected.

Change Section S4 to read as follows by the addition of the italicized sentence:

S4. The flattening test specified in Section 10 may be made on specimens from both ends of each length of pipe. *This requirement applies only to pipe over 2 in. in nominal diameter.* Crop ends may be used. If the specimen from either end of any length fails to conform to the specified requirements, that length shall be rejected.

In Section S5 delete the first sentence.

Delete Section S7 from the specifications.

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 39 T):

Section 2.—Change Paragraph (*d*) from its present form: namely,

(*d*) The steel melting practice shall be such as will produce a ferritic grain size in the finished pipe corresponding to an A.S.T.M. grain size of 3 to 6 under the following conditions: (1) in hot-finished pipe in the as-finished condition; (2) in bends and other fabricated parts which are heated and possibly water-quenched, if subsequently annealed at 1700 F. and slow-cooled in the furnace at a rate not to exceed 50 F. per hour until below 1000 F.

to read as follows:

(*d*) The steel melting practice shall be such as will produce a structural grain size[a] corresponding to an A.S.T.M. grain size of from 3 to 6 after the following specific heat treatment: heating to 1700 F. and holding approximately 1 hr. per inch of wall thickness, but in no case less than 1 hr., and cooling in the furnace at a rate of not to exceed 50 deg. Fahr. per hour until below 1000 F.

---

[a] The term "structural grain size" refers to the predominant grain as evidenced by ferrite grains, Widmänstatten areas, or pearlite patches.

Add a new Paragraph (*e*) to read as follows:

(*e*) Hot-finished pipe in the as-finished condition in nominal sizes 3 in. and over made to schedules 140 and 160 shall conform to the grain size requirements specified in Paragraph (*d*).

Section 4.—Add a new Paragraph (*b*) to read as follows, lettering the present section as Paragraph (*a*):

(*b*) Unless otherwise specified, cold-drawn pipe after finishing shall be given a process anneal by heating in a furnace from 1200 to 1300 F. followed by cooling in the furnace at a rate not to exceed 50 deg. Fahr. per hour until below 1000 F.

Section 8 (*a*).—Under the tabulation of tensile properties, change the footnote to read as follows by the addition of the italicized figures and the omission of those in brackets:

[a] For transverse tension tests the material shall conform to these minimum tensile requirements except that the elongation in 2 in. shall be [25] *20* per cent.

Section 11.—Make the same change in this section as recommended in Section 11 of Specifications A 158 - 38 T, above.

Section 13 (*c*).—Make the same change in this section as recommended in Section 12 (*c*) of Specifications A 158 - 38 T, above.

Section 14 (*a*).—Make the same change in this section as recommended in Section 14 of Specifications A 158 - 38 T, above.

Section 20 (*a*), (*b*) and (*c*).—Make the same changes in this section as recommended above in Section 19 (*a*), (*b*) and (*c*) of Specifications A 158 - 38 T.

Section 21.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

21. Each length of pipe manufactured in accordance with these specifications shall be legibly marked, either by stenciling, stamping, or rolling, with the manufacturer's private identifying mark; [together] with the symbols A 206 [and the alloy symbol (A 206 - P 1)] and an additional *symbol* S if the pipe conforms to the supplementary requirements specified in Paragraphs S1 to [S8] *S7; the length; the A.S.A. schedule number; and the heat number or manufacturer's number by which the heat can be identified.* Marking shall be within 12 in. of one end of each length. On pipe sizes 4 in. and larger, the weight shall be given. On small diameter pipe which is bundled, the above information may be legibly stamped on a metal tag securely attached to each bundle. When pipe marked as specified in this section is rejected, the designation A 206 shall be canceled. *All photomicrographs shall be properly identified as to heat number, and size and wall thickness of pipe from which the section was taken.*

Section 22 (*a*).—Change the last sentence to read as follows by the addition of the italicized words:

When specified in the order, the manufacturer shall notify the purchaser in time so that he may have his inspector present to witness any part of the manufacture or tests that may be desired.

Supplementary Requirements.—Make the same changes in Sections S1, S3, and S4 as recommended in these same sections of Specifications A 158 - 38 T, above.

Change Section S5 from its present form to read as follows:

S5. The steel shall have a homogeneous structure as shown by the etching tests described in Paragraph S6. The grain size of the steel shall conform to the requirements of Section 2 (*d*) except that in nominal pipe sizes 3 in. and over made to Schedules 140 and 160, each piece of pipe in the as-finished condition shall show a structural grain size corresponding to an A.S.T.M. grain size of 3 to 6 in accordance with the Tentative Classification of Austenite Grain Size in Steels (A.S.T.M. Designation: E 19) of the American Society for Testing Materials.

Add a sentence at the end of Section S7 to read as follows:

All photomicrographs required shall be properly identified as to heat number, size, and wall thickness of pipe from which the section was taken. Photomicrographs shall further be identified to permit association of each photomicrograph with the individual section of pipe it represents.

Delete Section S8 from the specifications.

Appendix I.—Change from its present form by deleting from the table the headings stating the pressure and temperature ratings.

Appendix II.—Immediately below the table add a note to read as follows:

NOTE.—This table of sizes and schedule numbers is included in the specifications for information only. Many of the sizes listed are not available in stock at pipe mills since they are not in common use and are difficult to obtain in small quantities. Wherever possible, it is recommended that the purchaser use the sizes and schedule numbers indicated in Appendix I, which were originally selected by the Prime Movers Committee of the Edison Electric Institute, and may, through increasing demand, eventually become standards for stock sizes.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Standard Specifications for Steel Plates of Structural Quality for Forge Welding (A 78 - 39):

Title.—Change from its present form, to read as follows:

Specifications for Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding.

Section 1.—Change from its present form, to read as follows:

1. These specifications cover two grades of carbon-steel plates of structural quality suitable for welding. They are suitable for forge welding when the requirements specified in Section 3 (*b*) are applied. The purchaser shall specify when the material is to be used for forge welding.

NOTE.—The term "forge welding" as used in these specifications refers to the process of heating with water gas, and hammering mechanically, without the use of fluxes.

Section 3.—In the table of chemical requirements in Paragraph (*a*) change the maximum sulfur content for grades A and B from "0.05 per cent" to read "0.055 per cent."

Change Paragraph (*b*) from its present form, to read as follows:

(*b*) When these specifications are used for material that is to be forge welded, the quantity of either nickel or chromium shall not exceed 0.05 per cent and, unless otherwise specified, the silicon shall not exceed 0.05 per cent.

Delete the note following this Section.

Section 10 (*a*).—Change from its present form to read as follows:

(*a*) Two tension and two bend tests shall be made from each melt, unless the finished material from a melt is less than 30 tons when one tension test and one bend test will be sufficient. If, however, material from one melt differs $\frac{3}{8}$ in. or more in thickness, one tension test and one bend test shall be made from both the thickest and the thinnest material rolled regardless of the weight represented.

Standard Specifications for Boiler Rivet Steel and Rivets (A 31 - 39):

Section 7.—Change from its present form to read as follows:

7. (*a*) *Cold-Bend Tests.*—The test specimen for grade A steel shall stand being bent cold through 180 deg. flat on itself without cracking on the outside of the bent portion.

(*b*) The test specimen for grade B steel shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion, as follows: For material $\frac{3}{4}$ in. and under in diameter, around a pin the diameter of which is equal to one-half the diameter of the specimen; for material over $\frac{3}{4}$ in. in diameter, around a pin the diameter of which is equal to the diameter of the specimen.

(*c*) *Quench-Bend Tests.*—The test specimen for grade A steel when heated to a light cherry red as seen in the dark (not less than 1200 F. (649 C.)), and quenched at once in water the temperature of which is between 80 and 90 F. (27 and 32 C.), shall stand being bent through 180 deg. flat on itself without cracking on the outside of the bent portion.

(*d*) The test specimen for grade B steel, when similarly heated and quenched, shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion, as follows: For material ¾ in. and under in diameter, around a pin the diameter of which is equal to the diameter of the specimen; for material over ¾ in. in diameter, around a pin the diameter of which is equal to one and one-half times the diameter of the specimen.

Section 14.—Change from its present form to read as follows:

14. (*a*) The rivet shank of grade A steel shall stand being bent cold through 180 deg. flat on itself, as shown in Fig. 1, without cracking on the outside of the bent portion.

(*b*) The rivet shank of grade B steel shall stand being bent cold through 180 deg. without cracking on the outside of the bent portion, as follows: For material ¾ in. and under in diameter, around a pin the diameter of which is equal to the diameter of the shank; for material over ¾ in. in diameter, around a pin the diameter of which is equal to one and one-half times the diameter of the shank.

Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 36):

Section 1 (a).—Change the last sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

When seamless pipe is ordered for close coiling, cold bending, or for forge welding [the low-carbon] grade *A seamless* should be specified rather than [the medium-carbon] grade *B seamless.*

This necessitates related editorial changes in Table I and Section 7.

Table I.—Change the minimum tensile strength requirement for grade B from "62,000 psi." to read "60,000 psi." Change the yield point requirement for grade A pipe from "26,500 psi." to read "30,000 psi."

New Section.—Add a new Section 17 covering Marking to read as follows, renumbering the remaining sections accordingly:

17. Each length of pipe shall be legibly marked by rolling, stamping, or stenciling to show the name or brand of the manufacturer; the type of pipe (that is, lap-welded, seamless A, or seamless B); XS for extra strong, XXS for double extra strong; ASTM A 53; the length, and the hydrostatic test pressure in pounds per square inch; except that for small diameter pipe which is bundled, this information may be marked on a tag securely attached to each bundle.

TABLE III.—HYDROSTATIC PRESSURES FOR WELDED AND SEAMLESS STEEL PIPE.[a]
(Pressures Expressed in Pounds per Square Inch)

Nominal Pipe Size, in.	"Standard Weight" Pipe			"Extra Strong" Pipe			"Double Extra Strong" Pipe		
	Butt-welded	Lap-welded and Grade A	Grade B	Butt-welded	Lap-welded and Grade A	Grade B	Butt-welded	Lap-welded and Grade A	Grade B
⅛ to 1, incl.	700	700[b,c]	700[c]	850	850[b,c]	850[c]	1000	1000[b,c]	1000[c]
1¼ to 3, incl.	800	1000	1100	1100	1500	1600	1200	1800	1900
3½ to 6, incl.	...	1200	1300	....	1700	1800	....	2000	2100
8	...	1200	1300	....	1700	2400	....	2800	2800
10 to 12, incl.	...	1000	1200	....	1600	1900	....	....	....

[a] For pipe over 12-in. in nominal pipe size, the test pressures should be calculated by the formula $P = \frac{2St}{D}$ in which P = pressure in pounds per square inch; S = fiber stress, 12,000 psi.; t = thickness of wall in inches; and D = outside diameter in inches.

[b] Lap-welded pipe is not made below 1¼-in. size.

[c] Seamless pipe in these small sizes will probably need to be cold drawn.

Table II.—Change from its present form to read as prescribed in the accompanying Table III.

Standard Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 36):

New Section.—Add a new Section 15 covering Marking to read as follows, renumbering the remaining sections accordingly:

15. Each length of pipe shall be legibly marked by rolling, stamping, or stenciling to show the

name or brand of the manufacturer, the length, and the hydrostatic test pressure in pounds per square inch; except that for small diameter pipe which is bundled, this information marked may be marked on a tag securely attached to each bundle.

Table I.—Make the same change in this table as recommended in Table II of Specifications A 53 - 36, above, adding the phrase "Electric-welded" after "Lap-welded" in the three subheadings in the table.

Standard Specifications for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 37):

Section 6.—Make the same change in this section as recommended in Section 6 of Specifications A 192 - 38 T.[1]

Section 8.—Make the same change in this section as recommended in Section 8 of Specifications A 214 - 39 T.[2]

Section 11 (b).—Make the same change in this section as recommended in Section 11 (*b*) of Specifications A 214 - 39 T.[2]

Standard Specifications for Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 - 38):

Section 7.—Make the same change in this section as recommended in Section 6 of Specifications A 192 - 38 T.[1]

Section 9.—Change from its present form to read as follows:

> 9. (*a*) Except as specified in Section 11 (*c*), the Rockwell hardness test shall be made on the inside of a specimen cut from the tube, but hardness tests shall not be required on tubes less than 0.065 in. in wall thickness.
>
> (*b*) The tubes shall have a Rockwell hardness number not to exceed B 85.
>
> (*c*) In making the Rockwell hardness test, reference should be made to the Standard Method of Test for Rockwell Hardness of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials.

Standard Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 39):

Table I.—Add the same requirements for a new chromium-molybdenum composition, designated grade 6, as recommended in Table I of Specifications A 213 - 39 T.[3]

Section 7.—Make the same change in this section as recommended in Section 6 of Specifications A 192 - 38 T.[1]

Section 9.—Make the same change in this section as recommended in Section 9 of Specifications A 187 - 38, above.

Standard Specifications for Seamless Low-Carbon Steel Still Tubes for Refinery Service (A 161 - 37):

Title.—Change the title of these specifications by the addition of the words "and Carbon-Molybdenum" after the words "Low-Carbon."

Section 1 (a).—Change to read as follows by the addition of the italicized words and the omission of the word in brackets:

> 1. (*a*) These specifications cover seamless, hot-rolled and cold-drawn, low-carbon *and carbon-molybdenum* steel still tubes [(Note)] 2 in. and over in outside diameter and thicker than No. 5 B.w.g. in minimum wall or $\frac{1}{4}$ in. in average wall, for use in carrying oil at elevated temperatures and pressures in various types of oil stills, in which the tubes may be subjected to a furnace temperature higher than that of the contained fluid.

Note.—Delete the note in Section 1 from the specifications.

Section 4 (a).—In the table of chemical composition add the following requirements for a new grade of carbon-molybdenum steel still tubes, labeling the present grade "Low-Carbon:"

---

[2] See p. 85.

[3] See p. 84.

Carbon, per cent	0.10 to 0.20
Manganese, per cent	0.30 to 0.60
Phosphorus, max., per cent	0.04
Sulfur, max., per cent	0.04
Silicon, per cent	0.10 to 0.50
Molybdenum, per cent	0.45 to 0.65

Section 6 (a).—In the tabulation of tensile properties add the following requirements for the new grade of carbon-molybdenum steel still tubes, labeling the present grade "Low-Carbon:"

Tensile strength, min., psi	55 000
Yield point, min., psi	30 000
Elongation in 2 in., min., per cent	30

Section 7.—At the end of this section add a new Paragraph (*c*) to read as follows:

(*c*) Superficial ruptures as a result of minor surface imperfections shall not be cause for rejection.

Section 9 (a).—Change the tabulation from its present form to read as follows by the addition of the italicized words and figures:

	Brinell Hardness Number	
	Low-Carbon	*Carbon-Molybdenum*
Hot-rolled tubes	137	*150*
Cold-drawn tubes	125	*137*

Table III.—Change the phrase "length of upset, in." to read "specified length of upset, in." with a footnote indicating that "specified length of full upset is minimum."

Change the note under the title of Table III to read:

NOTE.—For tubes that are upset at the ends, a "go" plug gage with a diameter determined by the appropriate formula given in the table shall enter each upset end the full length of the upset. A "no-go" plug gage using the same formula without the clearance allowance shall not enter the tube.

Section 14.—Change Paragraph (*d*) to read as follows by the addition of the italicized words:

(*d*) Permissible variations *in outside diameter* for hot-rolled tubes shall apply to the upset ends of both hot-rolled and cold-drawn tubes.

Add new Paragraphs (*e*) and (*f*) to read as follows:

(*e*) For upsets that increase the wall thickness more than $\frac{1}{16}$ in., the length at any point shall not be more than 50 per cent over that specified, or a minimum of $1\frac{1}{2}$ in. For upsets that increase the wall thickness $\frac{1}{16}$ in. or less, the length at any point shall not be more than $2\frac{1}{2}$ in. over that specified.

(*f*) For upset ends of tubes the wall thickness measured within $\frac{3}{4}$ in. from the finished end shall not exceed the actual average wall of that section by more than plus or minus 10 per cent. The actual average wall is defined as the average of the thickest and thinnest wall in that section.

Section 15 (a).—Change the third sentence from its present form to read as follows by the addition of the italicized words:

Minor defects may be removed by grinding, provided *that a smooth curved surface is maintained; that* the wall thickness is not decreased to less than that permitted in Section 15, *and such grinding shall not be limited by the permissible variations in outside diameter prescribed in Table I, except that for a distance 6 in. from each end of a tube the permissible variations in outside diameter shall be maintained.*

Standard Specifications for Seamless Alloy-Steel (4 to 6 per cent Chromium) Still Tubes for Refinery Service (A 188 - 38):

Section 7.—Make the same change in this section as that recommended in Section 7 of Specifications A 161 - 37, above.

Table III.—Make the same changes in this table as recommended above in Specifications A 161 - 37.

Section 15.—Make the same change in Paragraph (*d*) as recommended above in Section 14 (*d*) of Specifications A 161 - 37. Add new Paragraphs (*e*) and (*f*) as recommended above in Specifications A 161 - 37.

Section 16—Make the same change in Paragraph (*a*) of this section as recommended above in Section 15 (*a*) of Specifications A 161 - 37.

Change Paragraph (*b*) from its present form to read as follows:

(*b*) Tubes shall be free from scale, suitable for inspection.

Standard Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 39):

Table I.—Add the same requirements for a new chromium-molybdenum composition, designated Grade 6, as recommended in Table I of Specifications A 213 - 39 T.[3]

Section 7.—Make the same change in this section as recommended in Section 7 of Specifications A 161 - 37, above.

Table III.—Make the same changes in this table as recommended above in Specifications A 161 - 37.

Section 15.—Make the same change in Paragraph (*d*) as recommended above in Section 14 (*d*) of Specifications A 161 - 37. Add new Paragraphs (*e*) and (*f*) as recommended above in Specifications A 161 - 37.

Section 16.—Make the same change in Paragraph (*a*) of this section as recommended above in Section 15 (*a*) of Specifications A 161 - 37.

Make the same change in Paragraph (*b*) of this section as recommended in Section 16 (*b*) of Specifications A 188 - 38, above.

Standard Specifications for Commercial Quality Hot-Rolled Bar Steels (A 107 - 39):

Table I.—In grade 27, open-hearth resulfurized nut stock, change the carbon range from "0.15 to 0.25 per cent" to read "0.25 max. per cent" and the manganese range from "0.50 to 1.00 per cent" to read "0.40 to 0.90 per cent."

Spec. for Forged or Rolled Steel Pipe Flanges for High-Temperature Service (A 105 - 39):

Title.—Change from its present form to read as follows:

Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service.

Throughout the specifications, change the word "class" to read "grade."

Section 1 (*a*).—Change from its present form to read as follows:

(*a*) These specifications cover forged or rolled steel pipe flanges, forged fittings and valves and parts for high-temperature service (Note). The term "forgings" used in these specifications shall be understood to cover one or all of the products mentioned above, either forged or rolled.

Section 6.—Under the table of chemical requirements, change the footnote from its present form to read as follows:

[a] When flanges will be subject to fusion welding, the carbon content shall not exceed 0.35 per cent. When the carbon is restricted to 0.35 per cent max., it may be necessary to add silicon to the composition for grade II and for the heavier thicknesses of grade I flanges in order to meet the required tensile properties. The silicon content shall not exceed 0.30 per cent.

Section 10 (*c*).—Change from its present form to read as follows:

(*c*) For the purpose of making tension tests, the forging manufacturer shall supply suitable test specimens from the flange or, at his option, furnish test blanks prepared from the billets or forging bar entering into the finished product, provided such test blank has undergone approximately the same working and heat treatment as the finished product.

Standard Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for Service at Temperatures from 750 to 1100 F. (A 182 - 39):

Table I.—Change the carbon range for the 4–6 per cent chromium material from "0.15 to 0.25 per cent" to read "0.25 max. per cent."

Standard Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 – 39):

Table I.—Change from its present form to read as prescribed in the accompanying Table IV.

The deletion of grade C pipe from these specifications necessitates related editorial changes in the various sections referring to grade C pipe: namely, Sections 6, 7 (*c*), 17, and Note 2.

TABLE IV.—CHEMICAL REQUIREMENTS.

	Welded Open Hearth	Seamless	
		Grade A	Grade B Silicon-Killed
Carbon, max., per cent	....	0.25	0.35
Manganese, per cent	0.30 to 0.60	0.30 to 0.60	0.35 to 1.00
Phosphorus, max. per cent	0.045	0.04	0.04
Sulfur, max., per cent	0.06	0.06	0.06
Silicon, min., per cent	....	....	0.10

Table II.—Change the minimum tensile strength requirement for grade B from "62,000 psi." to read "60,000 psi."

Change the footnote to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> [a] For transverse tension tests the material shall conform to these minimum tensile requirements except that the elongation in 2 in. shall be [30] *25* per cent for grade A, *and* [20] *15* per cent for grade B. [and 15 per cent for grade C] seamless material.

Section 8.—Make the same changes in this section as recommended in Section 11 of Specifications A 158 – 38 T, above.

Section 9 (*c*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*c*) The transverse tension test may be made on seamless pipe 8 in. and over in nominal diameter. Specimens may be taken from a ring cut from the pipe or from sections resulting from the flattening tests [subsequently normalized]. The specimen shall consist of a strip cut transversely from the pipe; the width of the specimen shall be $1\frac{1}{2}$ in. and its gage length 2 in. When the pipe wall thickness exceeds $\frac{3}{4}$ in., the A.S.T.M. standard 2-in. gage length tension test specimen shown in Fig. 1 shall be used. Specimens cut from the ring section ***shall be flattened cold*** [may be flattened hot] and shall be parallel between gage marks. At the option of the manufacturer, the transverse tension test specimen may be machined off on either surface provided not over 15 per cent of the nominal thickness is removed from either side.

Section 11 (*a*).—Add a footnote reference after the word "lot" to read the same as recommended in Section 14 of Specifications A 158 – 38 T, above.

Section 16.—Make the same changes in Section 16 (*a*), (*b*), and (*c*) as recommended above in Section 19 (*a*), (*b*), and (*c*) of Specifications A 158 – 38 T.

Supplementary Requirements. — Change the first sentence in Section S1 to read as follows by the addition of the italicized words and the omission of the word in brackets:

> These requirements shall not be considered unless specified in the order, in which event *the following supplementary* [all] tests shall be made at the mill at the purchaser's expense and witnessed by his inspector before shipment of the material.

Make the same change in Section S5 as recommended above in Section S5 of Specifications A 158 – 38 T.

Delete Section S7 from the specifications.

Standard Specifications for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 – 36):

New Section.—Add a new Section 16 covering Marking to read as follows, renumbering the remaining sections accordingly:

> 16. Pressure containing castings, made in accordance with these specifications, shall be marked for identification with the melt number

or melt identification. Marking shall be in such position as not to injure the usefulness of the castings.

NOTE.—These marking requirements are taken from the Standard Marking System for Valves, Fittings, Flanges, and Unions (No. SP-25-1936) of the Manufacturers Standardization Society of the Valve and Fittings Industry.

Standard Specifications for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 – 39):

Section 1 (*b*).—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> [Nine] *Ten* grades are covered, including [seven] *eight* ferritic steels and two austenitic steels designated C 1, C 3A, etc.

Section 2.—Change the first sentence to read as follows by the addition of the italicized letters and figures:

> Steels C 1, C 3A, C 3B, C 4, C 5*A*, *C 5B*, and C 11 shall be made by the open-hearth, electric-furnace, or crucible process.

Section 3 (*c*).—Add the two new grades C 5A and C 5B to this section.

Table I.—Add the two new grades C 5A and C 5B, with requirements conforming exactly to the existing requirements for grade C 5, except that the maximum silicon content of C 5A will be "0.50 per cent" and for C 5B, "0.75 to 1.50 per cent."

Table II.—The physical requirements for grades C 5A and C 5B will be the same as those given for grade C 5.

Section 17.—Change from its present form to read as follows:

> 17. (*a*) Pressure containing castings, made in accordance with any of the grades of steel covered by these specifications, shall be marked for material identification with the A.S.T.M. symbols for the grade of steel, C1, C3a, etc., respectively and the melt number or melt identification. Marking shall be in such position as not to injure the usefulness of the castings.
>
> NOTE.—These marking requirements are taken from the Standard Marking System for Valves, Fittings, Flanges, and Unions (No. SP-25-1936) of the Manufacturers Standardization Society of the Valve and Fittings Industry.
>
> (*b*) Pressure containing castings, made of alloy steel grades not covered by these specifications, shall be marked as required in Paragraph (*a*) except that identifying symbols for the grade of steel shall be used which will not conflict with any A.S.T.M. symbols for steel identification.

Spec. for Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 39):

Table I.—Change the requirement for carbon for class 3 from "0.20 per cent, min." to read "0.35 per cent, max." and for molybdenum from "0.45 to 0.65 per cent" to read "0.40 to 0.60 per cent."

Section 10.—Change the last sentence to read as follows by the addition of the italicized words:

> The threads in the nut shall not strip when subjected to a stress equal to *130,000 psi. for class 1 and* 150,000 psi. *for classes 2, 2H, 3 and 4* figured from the mean diameter of the bolt.

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Structural Steel for Locomotives and Cars (A 113 – 39):

Section 11 (*a*).—Make the same change in this section as recommended in Section 10 (*a*) of Specifications A 78 – 39, above.

Standard Specifications for Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 – 39):

Section 8 (*c*).—Change from its present form to read as follows:

> (*c*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine, or by the use of dividers, or by an extensometer, at a crosshead speed not to exceed ⅛ in. per min. Where a definite yield point is not exhibited, the yield strength corresponding to a limiting permanent offset of 0.2 per cent of the gage length of the specimen shall be used

instead. The "offset method" of determining yield strength as described in the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials shall be followed. The tensile strength shall be determined at a speed of head not to exceed 1½ in. per min.

Standard Specifications for Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 - 36):

Section 8 (c).—Make the same change in this section as recommended in Section 8 (*c*) of Specifications A 27 - 39, above.

Standard Specifications for Alloy-Steel Castings for Structural Purposes (A 148 - 36):

Section 8 (b).—Make the same change in this section as recommended in Section 8 (*c*) of Specifications A 27 - 39, above.

Standard Specifications for Forged or Rolled Steel Pipe Flanges for General Service (A 181 - 37):

Section 5.—In the table of chemical composition change the manganese content for classes I and II from "0.40 to 0.80 per cent" to read "0.90 max. per cent."

Standard Specifications for Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 - 36):

Section 2.—Change to read as follows by the addition of the italicized words:

> 2. The steel shall be *a killed steel* made by either or both of the following processes: open-hearth or electric-furnace.

Table I.—Add a requirement for minimum silicon content for grades A, B, and C, to read "0.10 per cent."

Section 10 (c).—Add a new first sentence to this section to read as follows:

> If the percentage of elongation of any tension test specimen is less than that prescribed in Table II and any part of the fracture is outside the middle third of the gage length, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

Section 11 (a).—Add a footnote reference after the word "lot" to read the same as recommended in Section 14 of Specifications A 158 - 38 T, above.

Section 19.—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> *Each length of pipe which develops injurious defects during shop working or application operations will* [Material which shows injurious defects, subsequent to acceptance at the manufacturer's works or within one year after shipment from the manufacturer's works, may] be rejected, and the manufacturer shall be notified.

Supplementary Requirements.—Delete Section S6 from the specifications.

Explanatory Notes.—Delete Note 2 from the specifications.

ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard with the revisions as indicated below:

Tentative Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 38 T):

Section 5.—At the end of this section add Paragraph (*c*) to read as follows:

> (*c*) Superficial ruptures as a result of minor surface imperfections shall not be cause for rejection.

Tentative Specifications for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 38 T):

Section 6.—At the end of this section add a new Paragraph (*b*) to read as follows:

(*b*) Superficial ruptures as a result of minor surface imperfections shall not be cause for rejection.

Section 8.—Change from its present form to read as follows:

8. (*a*) For tubes 0.200 in. and over in wall thickness the Brinell hardness test shall be used and on tubes having wall thicknesses from 0.200 to 0.375 in., excl., a 10-mm. ball with 1500-kg. load, or a 5-mm. ball with 750-kg. load may be used, at the option of the manufacturer. For tubes less than 0.200 in. in wall thickness, the Rockwell hardness test shall be used.

(*b*) The Brinell hardness test may be made on the outside of the tube near the end or on the outside of a specimen cut from the tube, at the option of the manufacturer. The Rockwell hardness test shall be made on the inside of a specimen cut from the tube.

(*c*) The tubes shall have a hardness number not to exceed the following:

	Brinell Hardness Number (Tubes 0.200 in. and over in wall thickness)	Rockwell Hardness Number (Tubes less than 0.200 in. in wall thickness)
Hot-finished tubes..	137	B77
Cold-drawn tubes..	125	B72
Hot-finished tubes (annealed)......	125	B72

(*d*) In making the Brinell hardness test, reference should be made to the Standard Method of Test for Brinell Hardness of Metallic Materials (A.S.T.M. Designation: E 10) of the American Society for Testing Materials.

(*e*) In making the Rockwell hardness test, reference should be made to the Standard Method of Test for Rockwell Hardness of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials.

Section 11 (*b*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*b*) Five per cent of all tubes shall be selected [for the] *and a* hardness test *shall be made on each of these tubes as* specified in Section 8. [and one Brinell or one or more Rockwell tests shall be made on each of these tubes]

Tentative Specifications for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 - 38 T):

Section 7.—Make the same change in this section as recommended in Section 6 of Specifications A 192 - 38 T, above.

Section 9.—Change from its present form to read the same as recommended in Section 8 (*a*), (*b*), (*d*), and (*e*) of Specifications A 192 - 38 T, adding a sentence to Paragraph (*a*) to read as follows:

Hardness tests shall not be required on tubes less than 0.065 in. in wall thickness.

and also changing the last sentence of Paragraph (*b*) to read as follows:

Except as specified in Section 11 (*c*), Rockwell hardness tests shall be made on the inside of a specimen cut from the tube.

Incorporate the present hardness test values in a new Paragraph (*c*) to read as follows:

(*c*) The tubes shall have a Brinell hardness number not to exceed 143 and a Rockwell hardness number not to exceed B 79.

Section 12 (*e*).—Make the same change in this section as recommended in Section 11 (*b*) of Specifications A 192 - 38 T, above.

Tentative Specifications for Spiral-Welded Steel or Iron Pipe (A 211 - 38 T):

New Section.—Add a new Section 9 on Marking to read as follows, renumbering the remaining sections accordingly:

9. Each length of pipe shall be legibly marked with appropriate symbols by stenciling, stamping, or rolling, to show the name or brand of the manufacturer, the size, (diameter and wall thickness in inches), the hydrostatic test pressure in pounds per square inch, and the specification number A 211.

Tentative Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 39 T):

Table I.—Add the following requirements for a new chromium-molybdenum composition, designated T 22:

Carbon, max., per cent	0.15
Manganese, per cent	0.30 to 0.60
Phosphorus, max., per cent	0.03
Sulfur, max., per cent	0.03
Silicon, max., per cent	0.50
Chromium, per cent	2.00 to 2.50
Molybdenum, per cent	0.90 to 1.10

Table II.—Change to read as prescribed in the accompanying Table V by the addition of the italicized figures and the omission of the figures in brackets.

Section 7.—Make the same change in this section as recommended in Section 6 of Specifications A 192 – 38 T, above.

Section 9.—Change from its present form to read the same as recommended in Section 8 (*a*), (*b*), (*d*), and (*e*) of Specifications A 192 – 38 T, above, adding a sentence to Paragraph (*a*) to read as follows:

TABLE V.—CHEMICAL REQUIREMENTS OF AUSTENITIC STEEL.

Identification Symbol	T 8	T 18	T 19	T 20
Type	Chromium-Nickel	Chromium-Nickel-Titanium	Chromium-Nickel-Columbium	Chromium-Nickel-Molybdenum
Carbon, max., per cent	0.07	0.08	0.08	0.13
Manganese, per cent	[0.20 to 0.70] *2.00 max.*	[0.60 to 1.50] *3.00 max.*	[0.60 to 1.50] *3.00 max.*	[0.60 to 1.50] *3.00 max.*
Phosphorus, max., per cent	0.03	0.03	0.03	0.03
Sulfur, max., per cent	0.03	0.03	0.03	0.03
Silicon, max., per cent	0.75	[0.50] *0.75*	[0.50] *0.75*	0.60
Nickel, per cent	8.00 to [10.00] *11.00*	[10.00 to 12.00] *9.00 to 13.00*	[10.00 to 12.00] *9.00 to 13.00*	[12.50 to 14.50] *11.00 to 14.00*
Chromium, per cent	17.00 to [19.00] *20.00*	17.00 to [19.00] *20.00*	17.00 to [19.00] *20.00*	[15.50 to 17.00] *16.00 to 18.00*
Molybdenum, per cent	......	......	......	[2.50 to 3.25] *2.00 to 3.00*
Titanium, min., per cent	......	0.35	......	......
Columbium, min., per cent	......	......	0.70	......

> Hardness tests shall not be required on tubes less than 0.065 in. in wall thickness.

and also adding a sentence to Paragraph (*b*) to read as follows:

> Except as specified in Section 11 (*c*), Rockwell hardness tests shall be made on the inside of a specimen cut from the tube.

Incorporate the present hardness test values in a new Paragraph (*c*) to read as follows:

> The tubes shall have a Brinell hardness number not to exceed 163 for ferritic steel or 190 for austenitic steel, and a Rockwell hardness number not to exceed B 85 for ferritic steel or B 90 for austenitic steel.

Section 12 (*e*).—Make the same change in this section as recommended in Section 11 (*b*) of Specifications A 192 – 38 T, above.

Section 18 (*b*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*b*) The ferritic steel tubes shall be [commercially] free from scale, *suitable for inspection.* [The normal rotary straightening operation shall be deemed sufficient to remove scale for inspection.] If *special* pickling or shot-blasting [or both] are required, this shall be specifically stated in the order and shall be done at the purchaser's expense.

Tentative Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 39 T):

Section 6.—Make the same change in this section as recommended in Section 6 of Specifications A 192 – 38 T, above.

Section 8.—Change from its present form to read as follows:

> 8. (*a*) The Rockwell hardness test shall be made on the inside of a specimen cut from the tube, but hardness tests shall not be required on tubes less than 0.065 in. in wall thickness.
>
> (*b*) The tubes shall have a Rockwell hardness number not to exceed B 72.

(*c*) In making the Rockwell hardness test, reference should be made to the Standard Method of Test for Rockwell Hardness of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials.

Section 11 (*b*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*b*) Five per cent of all tubes shall be selected [for the] *and a* hardness test *shall be made on each of these tubes as* specified in Section 8. [and one Rockwell determination shall be made on each of these tubes]

Tentative Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 39 T):

Section 6.—Make the same change in this section as recommended in Section 6 of Specifications A 192 - 38 T, above.

Section 8.—Change from its present form to read the same as recommended in Section 8 (*a*), (*b*), (*d*), and (*e*) of Specifications A 192 - 38 T, above.

Incorporate the present hardness test values in a new Paragraph (*c*) to read as follows:

(*c*) The tubes shall have a Brinell hardness number not to exceed 125 and a Rockwell hardness number not to exceed B 72.

Section 11 (*b*).—Make the same change in this section as recommended in Section 11 (*b*) of Specifications A 192 - 38 T, above.

REPORT OF SECTIONAL COMMITTEE

ON

STANDARDIZATION OF DIMENSIONS AND MATERIALS OF WROUGHT IRON AND WROUGHT STEEL PIPE AND TUBING

ASA PROJECT: B36

No meeting of the Sectional Committee on Standardization of Wrought Iron and Wrought Steel Pipe and Tubing was held during the past year. However, a number of revisions in American standard specifications for various pipe materials which are a part of the American Standard for Wrought Iron and Wrought Steel Pipe (B 36.10 - 1939) were considered and approved by letter ballot of the sectional committee and subsequently approved by the A.S.A. during the past year. This is a continuance of the practice of keeping the American Standard for Pipe abreast of the art of pipe manufacture. The following standards were revised:

Standard Specifications for:

Welded Wrought-Iron Pipe (A 72 - 39; ASA B36.2-1939),
Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 - 39; ASA B36.4-1939),
Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 - 39; ASA B36.9-1939), and
Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 39; ASA B36.3-1939).

The standard for wrought-iron pipe has been revised to state more specifically that pipe shall be made from all pig puddled or processed wrought iron.

The specifications for electric-fusion-welded steel pipe both in the larger and smaller sizes were revised to include pipe welded by the straight seam or spiral seam process. Formerly, it was the intention to cover the straight-seam type only, although this was not explicitly stated. An additional revision concerned the requirements for the test specimen. It is now stated that the specimen for the tension and bend tests may, at the option of the purchaser, be taken perpendicularly across the weld.

The specifications for lap-welded and seamless steel pipe for high-temperature service received somewhat more extensive revision. The process clause was changed to require the use of a killed steel for seamless pipe in view of the trend in recent years to use a killed product where severe service is involved due to its superior high-temperature characteristics. Other revisions concerned the specification of hydrostatic test by formula instead of tabulating the requirements as before, a change in the flattening test for welded pipe to call for cold flattening of the specimen, the inclusion of heat treatment of the transverse tension test specimen when using a specimen resulting from the flattening test, or hot-flattening a ring section, and slight revisions in notes to Table I and weight requirements.

The continuing revision of the pipe standard is intended to keep pace with the industry and facilitate use of the standard in ordering pipe, with the view that ultimately all pipe will be ordered in accordance with the schedule series of the American standard.

As an editorial revision, Table 2 of ASA B36.10–1939 has been added as a supplement to the following:

Standard Specifications for:

Welded and Seamless Steel Pipe (A 53 - 36; ASA B36.1-1936),
Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 39; ASA B36.3-1939),
Electric-Resistance-Welded Steel Pipe (A 135 - 34; ASA B36.5-1935),
Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 - 39; ASA B36.9-1939),
Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service A 155 - 36; ASA B36.11-1939),
Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 36; ASA G8.7-1937),

Tentative Specifications for:

Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 - 38 T), and
Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 39 T).

Inclusion of Table 2 in these specifications as a supplement is an important step which, it is hoped, will aid materially in familiarizing the trade with the schedule series. Ultimate incorporation of Table 2 in the text of these specifications in place of the old tables of "standard weight," "extra strong," etc., will have to await the more widespread application of the pipe standard.

Every effort is being made by the officers of the committee to hasten acceptance of the standard by all concerned, particularly in the heating and plumbing trades where the members are less likely to be informed of recent American standards. Articles explaining the purpose of the standard and urging its use have appeared in a number of trade journals and magazines.

It is urged that everyone interested in any way in pipe should use whatever influence he may have to overcome the inertia which resists the wider acceptance and use of this American standard.

Respectfully submitted on behalf of the committee,

H. H. MORGAN,
Chairman.

SABIN CROCKER,
Secretary.

REPORT OF COMMITTEE A-3

ON

CAST IRON

Since the 1939 annual meeting of the Society, Committee A-3 on Cast Iron has held one meeting, in Detroit, Mich., on March 5, 1940. A second meeting will be held in June during this annual meeting of the Society.

Subsequent to the 1939 annual meeting, Committee A-3 presented to the Society through Committee E-10 on Standards, Tentative Specifications for Cast-Iron Pit-Cast Pipe for Water or Other Liquids (A 44 – 39 T), to replace immediately the Standard Specifications for Cast Iron Pipe and Special Castings (A 44 – 04), and Tentative Specifications for Cast Iron Soil Pipe and Fittings (A 74 – 39 T), to replace immediately Standard Specifications A 74 – 29. These recommendations were accepted[1] by Committee E-10 on August 24, 1939, and the tentative specifications appear in the 1939 Book of A.S.T.M. Standards.[2]

Tentative Specifications A 44 – 39 T were prepared by Sectional Committee A21 on Cast-Iron Pipe and Fittings, of which the Society is one of the sponsors, and the specifications have been approved by the American Standards Association as American standard (ASA No. A21.2–1939).

In the past year the committee has lost through death one of its most able, conscientious, and active members, R. S. MacPherran. Mr. MacPherran's membership on Committee A-3 dated from the organization of the committee in 1903. He served as chairman from 1926 to 1928, and was active on several subcommittees. The services that he rendered to Committee A-3 have been invaluable. Always a willing worker and cooperative in committee work, his loyalty and good fellowship will ever remain a memory. It is with most sincere regret that the committee records his death.

The present membership of the committee is 108, of which 29 are classified as producers, 44 as consumers, and 35 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. W. Bolton.
Vice-Chairman, J. T. MacKenzie.
Secretary, E. R. Young.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Pig Iron (H. M. Rich, chairman) has under preparation new specifications for pig iron, to replace the Standard Specifications for Foundry Pig Iron (A 43 – 24).

Subcommittee III on Cylinders (O. B. Schultz, chairman) plans to revise the Standard Specifications for Cast-Iron Locomotive Cylinders (A 45 – 14) in order to bring them up to date.

Subcommittee XVIII on Automotive Castings (A. L. Boegehold, chairman) is considering revising the Specifications for Automotive Gray-Iron Castings (A 159) to bring them into agreement with the Specifications for Cast Iron of the Society of Automotive Engineers.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee A-3 reported the following results of the letter ballot vote from a committee membership of 103: A 44 - 39 T, affirmative 42, negative 0, ballots marked "not voting" 22; A 74 - 39 T, affirmative 52, negative 1, ballots marked "not voting" 17.

[2] 1939 Book of A.S.T.M. Standards, Part I, pp. 960, 974.

I. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative specifications be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for:[3]

Automotive Gray-Iron Castings (A 159 - 35 T), and

Lightweight and Thin-Sectioned Gray-Iron Castings (A 190 - 36 T).

II. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

The committee recommends that the revision published as tentative in 1938[4] of the Standard Specifications for Gray-Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 – 30) be approved for reference to letter ballot of the Society for adoption as standard.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 108 members; 66 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked ["Not Voting"]
I. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Automotive Gray-Iron Castings (A 159 - 35 T).	47	0	19
Spec. for Lightweight and Thin-Sectioned Gray-Iron Castings (A 190 - 36 T).....	50	0	16
II. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Spec. for Gray-Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 - 30)....	53	0	13

This report has been submitted to letter ballot of the committee which consists of 108 members; 61 members returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. H. ROTHER,
Chairman.

H. C. AUFDERHAAR,
Secretary.

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[3] 1939 Book of A.S.T.M. Standards, Part I, pp. 957, 955.

[4] *Ibid.*, p. 1271.

REPORT OF COMMITTEE A-5

ON

CORROSION OF IRON AND STEEL

Committee A-5 on Corrosion of Iron and Steel has held two meetings during the past year: one on June 27, 1939, during the annual meeting of the Society in Atlantic City, N. J., and the other on March 7, 1940, at the spring group meetings of A.S.T.M. committees in Detroit, Mich.

Since the last report, 7 new members have been elected and 3 members have resigned; the committee membership now totals 128, of whom 57 are classified as producers, 43 as consumers, and 28 as general interest members.

I. NEW TENTATIVE STANDARD

On the recommendation of Subcommittee VII the committee recommends that a new Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles be accepted for publication as tentative.[1] This method is a combination of the Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Wire (A 191 – 38) and the Tentative Method of Test for Uniformity of Coating by the Copper Sulfate Dip Test (Preece Test) on Zinc-Coated (Hot-Galvanized) Steel Castings and Forgings, Gray Iron and Malleable Iron Castings (A 208 – 38 T) and includes revisions and changes that were necessary in order to make the method applicable to other materials, such as hardware and threaded materials. While the new method, when adopted as standard, is intended to replace the present Methods A 191 – 38 and A 208 – 38 T, the latter are being continued this year in their present status.

II. REVISIONS OF TENTATIVE STANDARDS

On the recommendation of Subcommittee VI, the committee recommends that the three tentative specifications covering electrodeposited coatings on steel be revised, as indicated in detail below, and continued as tentative. In general, the revisions provide for the inclusion of requirements for a thicker coating in the specifications for zinc coatings (A 164 – 39 T), and for nickel and chromium coatings (A 166 – 39 T), and an intermediate thickness of coating in the specifications for cadmium coating (A 165 – 39 T). It is expected that the revised specifications will more nearly fit the needs of the electroplating industry.

Tentative Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 – 39 T):[2]

Section 1.—Change the second sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> [Two] *Three* types of coatings (Notes 1 and 2) are covered: namely,
> *Type G.S.*
> Type L.S. [for general service, and]
> Type R.S. [for mild service.]

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[1] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 288.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 945.

Change Note 1 to read as follows by the addition of the italicized words and letters and the omission of those in brackets:

> NOTE 1: Explanation of Symbols.—The initial letters *G*, L, and R were adopted as arbitrary designations of grades of plating. The second letter (S) refers to steel as the base metal; [to provide for] other base metals [such as brass (B) and zinc (Z)] *are indicated by the letters B for brass, C for copper, and Z for zinc.*

In the first sentence of Note 2 add the term "type G.S." before the present reference to type L.S.

In the second paragraph of Note 2 replace the symbol "LS" with the symbol "G.S."

Section 3.—Add a new Paragraph (*a*) to read as follows, relettering the present Paragraphs (*a*) and (*b*) as Paragraphs (*b*) and (*c*), respectively:

> 3. (*a*) *Type G.S.*—On significant surfaces of the finished articles, the minimum thickness of type G.S. zinc coating shall be 0.001 in.

Transfer the Note following Section 3 to an appendix, designated Appendix I.

Appendix.—Designate the present appendix as Appendix II, and change the third paragraph to read as follows by the addition of the italicized words, figures, and letters and the omission of those in brackets:

> To deposit 0.001 in. of zinc with high efficiency in either acid or cyanide baths, it requires about 14 amp. hr. per sq. ft. To produce an average thickness of [0.00075] *0.0015* in., that is, 50 per cent more than the minimum thickness of [0.0005] *0.001* in. specified for type [LS] *G.S.*, it will, therefore, require about [11] *21* amp. hr. per sq. ft. This is equivalent to plating for 1 hr. at [11] *21* amp. per sq. ft. or to a corresponding period for any other current density. Similarly, *for type L.S. it will require about 11 amp. hr. per sq. ft. and* for type R.S. [it will require] about 3.5 amp. hr. per sq. ft. to deposit an average [of 0.00023 in. of zinc (or a minimum of 0.00015 in.)] *thickness about 50 per cent greater than the specified minimum thickness.*

Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel (A 165 – 39 T):[3]

Section 1.—Change the second sentence to read as follows by the addition of the italicized words and letters and the omission of the words in brackets:

> [Two] *Three* types of coating (Notes 1 and 2) are covered: namely,
> Type N.S. [for general service, and]
> *Type O.S.*
> Type T.S. [for mild service.]

Change Note 1 to read as follows by the addition of the italicized words and letters and the omission of those in brackets:

> NOTE 1: Explanation of Symbols.—The initial letters N, *O*, and T were adopted as arbitrary designations of grades of plating. The second letter (S) refers to steel as the base metal; [to provide for] other base metals [such as brass (B) and zinc (Z)] *are indicated by the letters B for brass, C for copper, and Z for zinc.*

In the first sentence of Note 2 add the term "type O.S." after the present reference to type N.S.

Section 3.—Add a new Paragraph (*b*) to read as follows, relettering the present Paragraph (*b*) as Paragraph (*c*):

> (*b*) *Type O.S.*—On significant surfaces of the finished articles, the minimum thickness of type O.S. cadmium coating shall be 0.0003 in.

Transfer the Note following Section 3 to an appendix, designated Appendix I.

Appendix.—Designate the present appendix as Appendix II, and change the last sentence of the third paragraph to read as follows by the addition of the italicized words, figures, and letters and the omission of those in brackets:

> Similarly, *for type O.S. it will require about 4.5 amp. hr. per sq. ft. and* for type T.S. [it

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[3] **1939 Book of A.S.T.M. Standards, Part I, p. 938.**

will require] about 2.5 amp. hr. per sq. ft. to deposit an average [of 0.00023 in. of cadmium (or a minimum of 0.00015 in.)] *thickness 50 per cent greater than the specified minimum thickness.*

Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 39 T):[4]

Section 1.—Change the second sentence to read as follows by the addition of the italicized words and letters and the omission of those in brackets:

> [Two] *Three* types of coating (Notes 1 and 2) are covered: namely,
> *Type F.S.*
> Type K.S. [for general service, and]
> Type Q.S. [for mild service.]

Change Note 1 to read as follows by the addition of the italicized words and letters and the omission of those in brackets:

> NOTE 1: Explanation of Symbols.—The initial letters *F*, K, and Q were adopted as arbitrary designations of grades of plating. The second letter (S) refers to steel as the base metal; [to provide for] other base metals [such as brass (B) and zinc (Z)] *are indicated by the letters B for brass, C for copper, and Z for zinc.*

In the first sentence of Note 2 add the term "type F.S." before the present reference to type K.S.; also in the second paragraph change "KS" to read "F.S."

New Section.—Add a new Section 3 covering "Thickness of Coatings, Type F.S.," to read as follows, renumbering the subsequent sections accordingly:

> 3. (*a*) *Nickel and Copper.*—For type F.S. coating on significant surfaces of the finished articles, the minimum thickness of the nickel coating or of the combined layers of copper and nickel shall be 0.00125 in., and if copper is used, the minimum thickness of the final nickel layer shall be 0.0006 in.
>
> (*b*) *Chromium.*—If a chromium finish is specified, the minimum thickness of the copper and nickel layers on significant surfaces shall be as specified in Paragraph (*a*), and the minimum thickness of the chromium coating shall be 0.00001 in.

Section 4.—Transfer the Note following Section 4 (new Section 5) to an appendix, designated Appendix I.

Section 9.—Change Paragraph (*a*) from its present form to read as follows:

> 9. (*a*) The plated articles shall show no appreciable corrosion on significant surfaces at the end of the following periods of continuous exposure to the salt spray test (Note):

Type F.S.	72 hr.
Type K.S.	48 hr.
Type Q.S.	16 hr.

To the Note following Section 9 (*b*) add the following summary of requirements for type F.S. coating:

Copper plus nickel, min.	0.00125 in.
Final nickel, min.	0.0006 in.
Chromium (if required), min.	0.00001 in.
Salt spray, continuous exposure	72 hr.

Appendix.—Designate the present appendix as Appendix II, and change the second sentence of the second paragraph from its present form to read as follows:

> General experience indicates that an average thickness at least 50 per cent greater than the specified minimum thicknesses of nickel plus copper will be required.

Under the heading "Copper and Nickel," change the last two sentences of the second paragraph from their present form to read as follows:

> For an average thickness of 0.0018 in. (type F.S.) it will, therefore, require about 25 × 1.8 = 45 amp. hr. per sq. ft.; for 0.0012 in. (type K.S.) about 30 amp. hr. per sq. ft.; and for 0.0006 in. (type Q.S.) about 15 amp. hr. per sq. ft.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 128 members; 95 members returned their ballots, with the results shown in Table I.

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[4] 1939 Book of A.S.T.M. Standards, Part I, p. 941.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles	80	4	11
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Electrodeposited Coatings of Zinc on Steel (A 164 - 39 T)	64	1	30
Spec. for Electrodeposited Coatings of Cadmium on Steel (A 165 - 39 T)	59	1	35
Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 39 T)	56	1	38

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Inspection of Annapolis Tests (E. S. Taylerson, chairman).—The specimens of No. 22 gage copper-bearing and non-copper-bearing corrugated black sheets that have been exposed at Annapolis, Md., since October 17, 1916, were inspected on April 18 and on October 16, 1939. At the April inspection it was found that three sheets had failed, namely, B2, U2, and V8. On October 16 it was found that sheets B3, E1, E2, E7, E9, H1, and T6 had failed. A summary of the failures to date is given in the accompanying Table II.

During the past year it was found necessary to move the exposure test at the Annapolis location to a new site because of the installation of new plant equipment by the Navy Department at the old location. Since the new site selected is adjacent to the old one, the atmospheric exposure conditions are substantially the same as before, and no marked difference in the corrosion rate of the samples is expected.

Subcommittee V on Total Immersion Tests (W. J. Jeffries, chairman).—The immersion tests in sea water in which the No. 16 and No. 22 gage sheet steel and wrought iron specimens and tubing samples were exposed at Portsmouth, N. H., and Key West, Fla., in 1927 have been concluded, with the failure of all the sheet specimens and the removal of the tube specimens from the test. The subcommittee is presenting its final report, appended hereto, on the sheet specimens characterizing the materials in the test, indicating the life of each specimen, and including such conclusions as the subcommittee has been able to draw.

Subcommittee V has been unable to complete the study of the pipe specimens under test but plans to submit its conclusions next year. Copper replicas of the riveted plates at Portsmouth, N. H., and Key West, Fla., were made during 1939, but no significant difference is noted since the last inspection. The riveted test plates immersed at Port Arthur, Tex., are reported to have been lost. The test at this location is therefore canceled and no report of the final results of the test will be possible.

Subcommittee VI on Specifications for Metallic-Coated Products (T. R. Galloway, chairman).—Only minor comments have been received on the Tentative Specifications for Zinc-Coated Steel Wire Strand, Galvanized and Extra Galvanized (A 122 - 39 T) and Class B and Class C Coatings (A 218 - 39 T), which were published as tentative last year; since the specifications have been available for such a short time, it is deemed advisable to retain them as tentative for another year.

The Subgroup on Chain-Link Fence Fabric has reported that revisions seem necessary in the present Standard Specifications for Chain-Link Fence Fabric Galvanized After Weaving (A 117 - 33) and that separate specifications for chain-link fence fabric galvanized before weaving appear desirable. It is hoped that agreement may be reached at the

TABLE II.—RECORD OF FAILURES IN ATMOSPHERIC EXPOSURE TESTS AT ANNAPOLIS OF NO. 22-GAGE COPPER-BEARING AND NON-COPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed October 17, 1916. Inspections Made Semiannually from 1917 to 1940.

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test[a]	Life of Each Failed Sheet in Years																Number of Sheets in Test[a]	Number of Sheets Failed	Average Life of Sheets in Years	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																					
Z.....	Open-hearth steel	B	0.032	0.32	0.063	0.042	.....	0.019	3	13.5	17.0	19.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	16.5	......Z
O.....	Open-hearth steel	A	0.121	0.536	0.008	0.030	0.249	0.020	12	7.0	9.0	9.0	9.0	9.0	9.5	10.0	10.0	11.0	11.0	13.0	17.0	..	..	..	..	12	12	10.4	......O
B.....	Open-hearth steel	A	0.09	0.38	0.017	0.062	0.036	0.084	3	20.0	20.5	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	2	....	......B
M....	Open-hearth steel	B	0.093	0.419	0.013	0.041	0.011	0.185	14	20.0	20.5	*	*	*	*	*	*	*	*	*	*	*	*	..	..	14	2	....	.....M
B.....	Open-hearth steel	A	0.06	0.27	0.014	0.053	0.006	0.187	9	19.5	20.0	21.5	22.0	22.5	23.0	*	*	*	..	..	..	..	..	..	..	9	6	....	......B
Z.....	Open-hearth steel	B	0.067	0.351	0.045	0.052	0.003	0.187	3	*	*	*					..	..	..	..	..	..	..	..	..	3	0	....	......Z
T.....	Open-hearth steel	B	0.083	0.313	0.009	0.020	0.005	0.227	14	19.5	19.5	19.5	21.5	21.5	21.5	23.0	*	*	*	*	*	*	*	..	..	14	7	....	......T
K.....	Open-hearth steel (acid)	A	0.107	0.447	0.091	0.046	0.004	0.237	16	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	16	0	....	......K
H.....	Open-hearth steel	A	0.069	0.387	0.016	0.027	0.004	0.244	11	19.5	21.0	21.5	23.0	*	*	*	*	*	*	*	..	..	..	..	..	11	4	....	......H
Z.....	Open-hearth steel	B	0.065	0.35	0.050	0.059	0.003	0.264	2	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	0	....	......Z
Z.....	Open-hearth steel	B	0.073	0.42	0.052	0.054	Trace	0.641	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	....	......Z
S.....	Open-hearth iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	7.0	8.0	8.5	9.0	9.5	10.0	11.0	11.5	11.5	12.5	12.5	12.5	12.5	12.5	..	..	14	14	10.6	......S
D....	Open-hearth iron	A	0.02	0.026	0.004	0.022	0.002	0.024	6	16.0	16.0	16.0	19.5	19.5	*									..	..	6	5	....	.....D
X....	Open-hearth iron	B	0.021	0.055	0.007	0.031	0.004	0.027	14	10.5	13.0	13.5	14.5	14.5	16.0	16.0	16.5	16.5	17.0	17.5	17.5	18.0	19.0	..	..	14	14	15.7	X
C.....	Open-hearth iron	A	0.01	0.028	0.002	0.027	0.002	0.141	3	17.5	20.5	*							..	..	..	..	..	..	..	3	2	...	C
C.....	Open-hearth iron	A	0.015	0.028	0.006	0.036	0.003	0.194	9	16.5	16.5	18.5	19.0	19.0	20.0	20.5	21.5	21.5	..	..	..	..	..	..	..	9	9	19.2	C
U.....	Open-hearth iron	B	0.017	0.074	0.008	0.037	0.005	0.260	13	15.5	16.5	19.0	19.0	19.0	19.0	19.0	19.5	20.0	20.0	21.0	21.0	22.5	..	..	..	13	13	19.3	......U
V.....	Open-hearth iron	B	0.018	0.051	0.007	0.021	0.005	0.304	14	17.0	17.0	17.5	18.5	18.5	19.0	20.0	20.0	20.5	21.0	22.0	22.0	22.5	*	..	..	14	13	....	V
A.....	Bessemer steel	A	0.038	0.386	0.089	0.040	0.007	0.014	15	15.5	16.5	16.5	17.0	17.0	17.0	18.0	19.0	19.0	19.5	19.5	19.5	19.5	19.5	20.0	..	15	15	18.2	......A
Z.....	Bessemer steel	B	0.063	0.41	0.102	0.044	0.009	0.013	3	11.5	11.5	11.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	11.5	......Z
Z.....	Bessemer steel	B	0.077	0.44	0.095	0.036	0.003	0.140	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	....	......Z
I.....	Bessemer steel	A	0.041	0.365	0.097	0.068	0.008	0.252	16	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	16	0	....	I
Z.....	Bessemer steel	B	0.058	0.37	0.093	0.043	0.009	0.269	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	....	......Z
Z.....	Bessemer steel	B	0.055	0.38	0.110	0.046	.....	0.527	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	....	......Z
Y.....	Wrought iron	B	0.03	0.055	0.139	0.021	0.218	0.020	3	18.0	19.5	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	2	....	......Y
E.....	Wrought iron	A	0.033	0.034	0.114	0.021	0.134	0.283	11	19.5	20.5	21.0	21.0	23.0	23.0	23.0	23.0	*	*	*	..	..	..	..	..	11	8	....	......E
							Total..........		220																Total....	220	134		

* Indicates unfailed sheets.

[a] The number of sheets in the test indicated in this table is less in some instances than the number shown in similar tables in earlier reports because certain sheets were discarded due to mechanical damage.

June meeting on the commercial difficulties which have been delaying the preparation of these specifications.

The Subgroup on Galvanized Sheets has not yet reached a conclusion in its attempt to eliminate differences between the A.S.T.M. and Federal specifications covering galvanized sheets. Specifications A 93 - 38 T will, therefore, be retained as tentative for another year.

The Subgroup on Electroplated Coatings on Steel has proposed the addition of a weight-of-coating class to each of the three tentative specifications sponsored by it (A 164, A 165, and A 166). The detailed revisions recommended in these specifications are given earlier in this report.

At the March meeting there was a discussion of the Tentative Specifications for Zinc Coating on Hardware and Fastenings (A 153 - 33 T), resulting in the conclusion that a single specification is more desirable than several specifications covering related products and that more than one weight-of-coating class seems desirable. It is expected that a subcommittee will be organized before the June meeting to proceed with this work.

Inquiries regarding the low aluminum limit prescribed in the Standard Specifications for Zinc Coatings on Structural Steel (A 123 - 33) have been discussed and were referred by the advisory committee to Subcommittee VIII to determine whether or not the allowable percentage of aluminum may be increased with safety. In the meantime, these specifications will remain unchanged.

Subcommittee VII on Methods of Testing (R. W. Baker, chairman).—Following up its favorable report on the possibility of combining the Methods of Test for Uniformity of Coating by the Preece Test on Zinc-Coated Iron or Steel Wire (A 191 - 38) and on Steel Castings and Forgings, Gray Iron and Malleable Iron Castings (A 208 - 38 T), the Subgroup on the Preece Test is presenting for publication as tentative, as mentioned earlier in this report, a combined method[1] which includes revisions, and additions to the method to cover the performance of this test on hardware and threaded materials. Standard Method A 191 - 38 and Tentative Method A 208 - 38 T will be continued in their present status until the new method is adopted as standard.

Based on investigations of the Joint Committee on Exposure Tests of Plating, composed of representatives of the American Electro-Platers' Society, the National Bureau of Standards, and the A.S.T.M., it is proposed to revise the Tentative Methods of Test for Local Thickness of Electrodeposited Coatings on Steel (A 219 - 39 T), to include in the procedure for determining the thickness of chromium, a graph showing the temperature factor for hydrochloric acid of 1.18 sp. gr. in addition to the curve now included which shows the temperature factor for hydrochloric acid of 1.19 sp. gr. This matter has been referred back to the Joint Committee and no final action will be taken until approval is received from the Joint Committee.[5]

Subcommittee VIII on Field Tests for Metallic Coatings (R. F. Passano, chairman).—There are three field tests now in progress: (*1*) on galvanized sheets, (*2*) on metallic coated hardware, structural shapes, tubular goods, etc., and (*3*) on wire and wire products. The report this year, which deals principally with the galvanized sheets, is appended. The atmospheric exposure tests on hardware and wire are scheduled for extensive consideration next year but a brief interim report on the wire test follows:

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[5] See Editorial Note, p. 97.

Inspections made since the last published report[6] show further rusting of the lighter weight zinc-coated wires at Pittsburgh, Pa., Sandy Hook, N. J., and Bridgeport, Conn. No rusting has taken place in the groups carrying coatings heavier than 1.0 oz. per sq. ft. at Pittsburgh or heavier than 0.45 oz. per sq. ft. at Sandy Hook. At State College, Pa., and at the remaining seven sites no rust has appeared on any coated wires.

The samples exposed for the determination of loss of tensile strength are being periodically removed and tested. Final trends of strength loss tests will be published when sufficient data have been assembled.

Concerning the zinc-coated specimens that were exposed for the purpose of periodic removal in order to determine the loss of weight of coating, the status is as follows: All samples have been removed from the Pittsburgh test site and some have been removed from Sandy Hook and State College. The annual losses of weight now indicated are: 0.369 oz. per sq. ft. for Pittsburgh; 0.119 oz. per sq. ft. for Sandy Hook; and 0.071 oz. per sq. ft. for State College. Specimens to determine the loss of weight of coating were not exposed at any of the other sites.

The following officers of Committee A-5 have been elected for the ensuing term of two years:

Chairman, W. H. Finkeldey
First Vice-Chairman, W. G. Kelley
Second Vice-Chairman, L. W. Hopkins

Mr. T. R. Galloway has accepted reappointment as secretary of the committee, and the present subcommittee chairmen have been reappointed.

This report has been submitted to letter ballot of the committee which consists of 128 members; 95 members returned their ballots, of whom 80 have voted affirmatively and 3 negatively.

Respectfully submitted on behalf of the committee,

W. H. FINKELDEY,
Chairman.

T. R. GALLOWAY,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee A-5 presented to the Society through Committee E-10 on Standards a revision of the Tentative Methods of Test for Local Thickness of Electrodeposited Coatings on Steel (A 219–39 T). This revision was accepted by Committee E-10 on November 5, 1940, and the methods in their revised form appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 303.

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 156 (1939).

REPORT OF SUBCOMMITTEE V ON TOTAL IMMERSION TESTS

The No. 22 gage and No. 16 gage black sheet steel and wrought iron specimens subjected to total immersion in sea water at Portsmouth, N. H., and Key West, Fla., in 1927 have all failed, and after studying the data obtained, the subcommittee has come to certain conclusions. These conclusions and the data in Tables I to IV are submitted herewith. Before proceeding with the report the subcommittee considers it advisable to explain the method employed in arranging the specimens for the test.

Each sheet included in the atmospheric exposure investigation under the cognizance of Subcommittee III, of Committee A-5, had one or more 2-in. wide strips sheared from it. These strips were stored at the National Bureau of Standards, Washington, D. C., from 1916 to 1920, when a majority of the strips were sheared to specimens measuring 2 in. wide by 6 in. long for use in the immersion tests in Calumet mine water, tap water at Washington, D. C., and Severn River water at Annapolis, Md., under the cognizance of Subcommittee V. The balance of the strips were sheared to similar size for immersion in sea water at Portsmouth, N. H. and Key West, Fla.

Twenty-five grades of metal were found to remain in the No. 22 gage and twenty-three grades in the No. 16 gage. The 2 by 6-in. specimens were pickled and weighed before being immersed in the sea water. The original weights cannot now be found in the records of the subcommittee. Each grade of metal was represented by six specimens each about 2 by 6 in. in each gage.

Two boxes were provided at each location, one of which contained the No. 22 gage and the other contained the No. 16 gage specimens. In each box there were six racks with slots to hold the specimens at both ends. The pieces were so arranged in the racks that in no one box would they be in the same order. The flow of water at the inlet was to be regulated so that approximately 6 gal. per min. entered the box at one end and flowed out at the opposite end.

The criterion of failure was complete perforation of the specimen, or an indentation of the edge at least $\frac{1}{4}$ in. by corrosion. A perforation occurring where the specimen was marked was in no case to be considered as a failure, it being assumed that the deformed metal at this location could be expected to fail earlier than the unstressed areas.

When the test had been in progress for 30 days, rack No. 1 was taken out and the specimens cleaned free from rust with a light bristle brush, but were not weighed. No failures having occurred, the rack was put back in place. At the end of the second month rack No. 2 was taken out and the specimens cleaned with the light brush. No failures having occurred, the specimens were returned to the rack and replaced in the box. The same procedure was followed with the other four racks at one month intervals, and on the seventh month rack No. 1 was again examined, and the same procedure followed until the first failure occurred, at which time and each month thereafter, all the racks were taken out, each specimen cleaned and all returned to the racks except the failed specimens.

It will be noted that after the first failure occurred, all specimens were subjected to the continuous immersion for one month. Up to the time the first failure was discovered, neglecting the first round of inspection, the test was a continuous immersion test for six months. For the first round of inspection, rack No. 1 was continuously immersed for 1 month, followed by a second continuous immersion for 6 months. Rack No. 2 was continuously immersed for 2 months followed by a continuous immersion for 6 months. Rack No. 3 was continuously immersed for 3 months followed by continuous immersion for 6 months. Rack No. 4 was continuously immersed for 4 months followed by continuous immersion for 6 months. Rack No. 5 was continuously immersed for 5 months followed by continuous immersion for 6 months. Rack No. 6 was continuously immersed for 6 months followed by continuous immersion for 6 months. This variation of time of continuous immersion of the several racks may have some bearing on what was later found.

From other immersion tests the subcommittee has learned that the loss of weight from a test specimen is greater the first month than it is the second, and greater the second month than it is the third, and so on.

The above schedule of inspection was continued until the first failure occurred. Each time an inspection was conducted, the specimens were brushed with a light bristle brush. The first failures occurred at Portsmouth, N. H., and at Key West, Fla., after 13 months. No attempt was made to carry the cleaning beyond the light brushing until a total of 22 months had elapsed. During the 22 months the specimens at Key West had accumulated a growth of coral which clung tenaciously to the surface of the specimens so long as the specimen was but slightly deformed by flexure. The coral growth broke off slightly in some places leaving the underlying metal bare.

In January, 1929, E. S. Taylerson, R. F. Passano and F. N. Speller were present during the regular monthly inspection of the specimens at Key West. Close examination of the specimens revealed failures in spots that had lost their coral growth. Up to that time failures had occurred where coral growth had been continuous. In this respect the conditions incident to failure had apparently changed at some intervening period, as the specimens having a continuous coating of coral intact over the entire surface seemed in no danger of ever failing.

During this inspection it was suggested that it was the custom in that particular locality to clean, by sandblasting, the bottoms of boats once a year. It was therefore agreed to follow this practice and the specimens were each cleaned by light sandblasting in order to equalize the surface condition of each specimen before continuing the test. It was also decided to sandblast each unfailed specimen once each year at Key West and at Portsmouth every 6 months.

Other than the fact that the specimens had been pickled prior to the beginning of the test and after 1929 were sandblasted, the method followed in the inspection each month did not differ from that used on the three previous immersion tests which are described in the 1920 Report of Committee A-5.[1]

It will be noted that when the first failure was discovered in any box, which contained six racks, all the racks in that box were removed and inspected each month thereafter, beginning with that

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part I, p. 227 (1920).

TABLE I.—FINAL RECORD OF FAILURES IN TOTAL IMMERSION TESTS AT PORTSMOUTH, N. H., OF NO. 22 GAGE BLACK SHEETS IMMERSED IN SEA WATER

Specimens Exposed March 7, 1927. Inspections Made Monthly from 1927 to 1936.

Group	Designation	Series	Average Analysis, per cent						Failures, days						Average Life in Days	Group
									Rack Number							
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	1	2	3	4	5	6		
O.....	Open-hearth steel	A	0.142	0.545	0.007	0.027	0.217	0.015	399	761	669	669	786	761	674	......O
Z4....	Open-hearth steel	B	0.100	0.319	0.069	0.044	....	0.018	819	973	1177	1003	819	1123	986	......Z4
F.....	Open-hearth steel	A	0.293	0.503	0.026	0.039	0.010	0.038	1003	786	669	786	1123	1284	942	......F
B.....	Open-hearth steel	A	0.110	0.380	0.016	0.065	0.013	0.082	*	....	819	973	1003	973	942	......B
B.....	Open-hearth steel	A	0.090	0.210	0.009	0.060	0.004	0.190	*973	913	....	....	....	....	943	......B
M.....	Open hearth steel	B	0.090	0.400	0.011	0.039	....	0.187	464	913	669	786	669	728	705	......M
Z5....	Open-hearth steel	B	0.060	0.360	0.047	0.056	....	0.198	940	1003	1066	1123	786	1241	1027	......Z5
T.....	Open-hearth steel	B	0.082	0.309	0.009	0.022	....	0.214	913	761	1036	819	669	1003	867	......T
G.....	Open-hearth steel	A	0.290	0.510	0.027	0.039	0.010	0.241	464	1066	464	1123	728	1151	833	......G
H.....	Open-hearth steel	A	0.063	0.403	0.017	0.027	0.005	0.245	464	786	399	669	428	1177	654	......H
Z6....	Open-hearth steel	B	0.075	0.390	0.053	0.053	Trace	0.261	728	973	1318	940	1264	1400	1104	......Z6
S.....	Open-hearth iron	B	0.016	0.020	0.006	0.023	....	0.018	669	913	940	973	786	1066	891	......S
D.....	Open-hearth iron	A	0.033	0.025	0.004	0.020	....	0.025	973	464	910	855	1003	819	842	......D
X.....	Open-hearth iron	B	0.016	0.054	0.006	0.032	....	0.025	761	973	1036	1090	1066	1177	1017	X
C.....	Open-hearth iron	A	0.032	0.025	0.005	0.030	0.006	0.178	884	973	819	1177	669	1066	931	C
U.....	Open-hearth iron	B	0.012	0.066	0.008	0.039	....	0.258	973	973	819	1003	761	1036	928	......U
V.....	Open-hearth iron	B	0.017	0.048	0.006	0.021	....	0.302	1003	973	1318	1003	761	1066	1021	V
A.....	Bessemer steel	A	0.038	0.380	0.094	0.042	0.007	0.012	1177	1003	1123	1090	1284	1459	1189	......A
Z.....	Bessemer steel	B	0.061	0.400	0.114	0.048	....	0.012	1123	973	1036	1003	1284	1066	1081	......Z
Z1....	Bessemer steel	B	0.071	0.393	0.089	0.045	0.009	0.135	786	1123	1123	1318	1123	1318	1132	......Z1
K.....	Acid open-hearth steel	A	0.102	0.455	0.087	0.045	....	0.238	1003	1066	1003	1431	1318	1490	1219	......K
I.....	Bessemer steel	A	0.041	0.382	0.098	0.068	....	0.250	973	1066	1090	1090	1151	1241	1105	I
Z2....	Bessemer steel	B	0.050	0.380	0.089	0.037	0.005	0.259	913	1123	1431	1241	973	1090	1129	......Z2
Z3....	Bessemer steel	B	0.041	0.365	0.095	0.045	....	0.518	940	1284	1036	1264	1400	1431	1226	......Z3
Y.....	Wrought Iron	B	0.030	0.058	0.154	0.020	0.223	0.021	973	1003	1123	819	1003	1003	987	......Y
E.....	Wrought Iron	A	0.024	0.034	0.111	0.022	0.146	0.297	913	884	1003	1177	1003	973	992	......E
					Average for each rack............				844.9	959.0	967.0	1014.7	952.4	1132.1		

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* Omitted from average for racks.

TABLE II.—FINAL RECORD OF FAILURES IN TOTAL IMMERSION TESTS AT KEY WEST, FLA., OF NO. 22 GAGE BLACK SHEETS IMMERSED IN SEA WATER.

Specimens Exposed March 1, 1927. Inspections Made Monthly from 1927 to 1938.

Group	Designation	Series	Average Analysis, per cent						Failures, days						Average Life in Days	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Rack Number							
									1	2	3	4	5	6		
O.....	Open-hearth steel	A	0.118	0.522	0.009	0.029	0.223	0.021	693	381	462	381	693	1379	645	......O
Z4....	Open-hearth steel	B	0.042	0.318	0.063	0.042	....	0.019	462	381	462	1317	891	462	663	......Z4
F.....	Open-hearth steel	A	0.293	0.503	0.026	0.039	0.010	0.038	462	679	1990	1194	891	1410	1104	......F
B.....	Open-hearth steel	A	0.023	0.229	0.012	0.052	0.004	0.117	*	1657	693	1627	....	....	1326	......B
B.....	Open-hearth steel	A	0.038	0.232	0.009	0.063	0.004	0.167	*1379	....	....	....	952	1657	1329	......B
M.....	Open-hearth steel	B	0.097	0.396	0.014	0.043	0.012	0.184	557	952	1317	891	1410	1317	1074	.....M
Z5....	Open-hearth steel	B	0.067	0.351	0.045	0.052	....	0.187	693	891	891	983	891	1044	899	......Z5
T.....	Open-hearth steel	B	0.078	0.322	0.010	0.020	....	0.237	1286	462	1627	1471	1349	1349	1257	......T
G.....	Open-hearth steel	A	0.290	0.510	0.027	0.039	0.010	0.241	493	1256	1194	1256	1286	1750	1206	......G
H.....	Open-hearth steel	A	0.076	0.371	0.011	0.028	0.002	0.246	983	693	462	1410	1194	1134	979	.....H
Z6....	Open-hearth steel	B	0.062	0.388	0.054	0.053	....	0.275	462	891	891	891	952	891	830	......Z6
S.....	Open-hearth iron	B	0.009	0.016	0.007	0.020	0.008	0.022	891	1078	1164	1349	1627	1780	1315	......S
D.....	Open-hearth iron	A	0.012	0.027	0.003	0.022	0.002	0.021	738	1657	1780	1502	921	1471	1345	.....D
X.....	Open-hearth iron	B	0.010	0.065	0.007	0.026	....	0.033	618	381	1502	1410	1044	1596	1092	X
C.....	Open-hearth iron	A	0.025	0.034	0.006	0.039	0.020	0.198	1078	1780	1780	693	1225	1720	1379	C
U.....	Open-hearth iron	B	0.016	0.075	0.009	0.034	0.003	0.269	1410	1103	1349	1410	1533	1720	1421	......U
V.....	Open-hearth iron	B	0.012	0.048	0.008	0.020	....	0.299	618	1349	1750	1564	1720	1750	1459	V
A.....	Bessemer steel	A	0.038	0.380	0.090	0.036	....	0.017	618	983	427	1164	856	1078	854	......A
Z.....	Bessemer steel	B	0.062	0.405	0.100	0.043	....	0.014	462	679	693	588	1012	891	721	......Z
Z1....	Bessemer steel	B	0.075	0.449	0.092	0.035	....	0.140	557	647	693	462	693	693	624	......Z1
K.....	Acid open-hearth steel	A	0.112	0.438	0.090	0.044	0.002	0.231	381	427	952	983	693	1256	782	.....K
I.....	Bessemer steel	A	0.041	0.372	0.094	0.067	0.010	0.254	462	1256	462	462	679	891	702	I
Z2....	Bessemer steel	B	0.058	0.370	0.093	0.043	....	0.266	829	829	693	829	891	1256	888	......Z2
Z3....	Bessemer steel	B	0.055	0.380	0.111	0.044	....	0.522	693	891	1012	1134	1194	1349	1046	......Z3
Y	Wrought Iron	B	0.039	0.052	0.146	0.021	0.213	0.019	557	693	462	647	381	891	605	......Y
E.....	Wrought Iron	A	0.039	0.032	0.114	0.021	0.110	0.241	891	618	1164	952	1078	921	937	......E
					Average for each rack...........				...703.9	873.2	1049.1	1039.3	1046.0	1250.0		

* Omitted from average for racks.

TABLE III.—FINAL RECORD OF FAILURES IN TOTAL IMMERSION TESTS AT PORTSMOUTH, N. H., OF NO. 16 GAGE BLACK SHEETS IMMERSED IN SEA WATER.

Specimens Exposed March 7, 1927. Inspections Made Monthly from 1927 to 1936.

Group	Designation	Series	Average Analysis, per cent						Failures, days						Average Life in Days	Group
									Rack Number							
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	1	2	3	4	5	6		
OO....	Open-hearth steel	A	0.117	0.377	0.010	0.027	0.005	0.030	1090	2221	1765	1643	1978	1796	1749	.....OO
ZZ4...	Open-hearth steel	B	0.028	0.392	0.042	0.029	....	0.028	1431	2374	1947	1765	2710	2678	2151	......ZZ4
FF....	Open-hearth steel	A	0.277	0.500	0.027	0.038	0.010	0.034	1673	2586	2374	2101	2861	3107	2228	.....FF
MM....	Open-hearth steel	B	0.105	0.420	0.011	0.032	0.005	0.052	2009	3045	2404	2831	2374	3107	2628	....MM
BB....	Open-hearth steel	A	0.063	0.338	0.030	0.057	0.078	0.122	1734	2922	3257	2770	3287	3195	2861	.....BB
GG....	Open-hearth steel	A	0.293	0.506	0.028	0.039	0.010	0.217	2586	2618	2922	3133	2922	3107	2881	.....GG
TT....	Open-hearth steel	B	0.076	0.305	0.010	0.020	....	0.226	1613	3163	2374	2831	3107	2710	2633	.....TT
HH....	Open-hearth steel	A	0.073	0.372	0.015	0.027	....	0.228	1066	3012	2101	2741	2101	2922	2324	.....HH
ZZ5...	Open-hearth steel	B	0.066	0.373	0.056	0.066	....	0.227	2710	2741	2618	3287	3045	2528	2822	......ZZ5
SS....	Open-hearth iron	B	0.014	0.017	0.006	0.023	....	0.028	2131	2555	2801	2678	2586	2528	2547	......SS
DD....	Open-hearth iron	A	0.020	0.034	0.004	0.026	....	0.028	1151	2586	1497	2678	1009	1734	1776	.....DD
XX....	Open-hearth iron	B	0.019	0.053	0.008	0.031	....	0.027	1765	1978	2710	2893	2555	2586	2415	XX
CC....	Open-hearth iron	A	0.020	0.031	0.044	0.033	....	0.065	1151	2678	2770	2221	2586	2893	2383	CC
CCC	Open-hearth iron	A	0.018	0.027	0.005	0.027	0.005	0.221	2009	3195	3287	3287	3195	3439	3069	CCC
UU....	Open-hearth iron	B	0.013	0.066	0.010	0.033	....	0.243	2648	3287	2922	3257	2922	3227	3044	.....UU
VV....	Open-hearth iron	B	0.016	0.033	0.007	0.022	....	0.289	2374	3195	3257	2922	3473	3195	3069	VV
AA....	Bessemer steel	A	0.053	0.420	0.087	0.041	....	0.013	2009	2374	2922	3045	2922	2951	2704	.....AA
ZZ....	Bessemer steel	B	0.078	0.412	0.100	0.038	....	0.013	2191	2922	2922	2922	2586	2922	2744	......ZZ
KK....	Acid open-hearth steel	A	0.120	0.418	0.085	0.042	....	0.240	2009	3045	2282	2922	2741	2831	2638	.....KK
II....	Bessemer steel	A	0.057	0.390	0.098	0.066	....	0.247	2586	2922	2741	2922	2861	3227	2877	II
ZZ2...	Bessemer steel	B	0.088	0.317	0.092	0.045	0.000	0.276	2801	3107	2861	3163	2831	2528	2882	......ZZ2
YY....	Wrought Iron	B	0.030	0.052	0.123	0.018	0.193	0.024	1400	2465	1673	2555	2374	2465	2155	.....YY
EE....	Wrought Iron	B	0.030	0.040	0.124	0.018	0.130	0.334	1734	2345	2801	3287	2801	2922	2648	.....EE
					Average for each rack...........				1907.4	2753.7	2574.3	2776.3	2688.1	2808.6		

TABLE IV.—FINAL RECORD OF FAILURES IN TOTAL IMMERSION TESTS AT KEY WEST, FLA., OF NO. 16 GAGE BLACK SHEETS IMMERSED IN SEA WATER.

Specimens Exposed March 7, 1927. Inspections Made Monthly from 1927 to 1938.

Group	Designation	Series	Average Analysis, per cent						Failures, days						Average Life in Days	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Rack Number							
									1	2	3	4	5	6		
OO....	Open-hearth steel	A	0.110	0.360	0.008	0.029	Trace	0.026	982	2143	2761	2698	1409	3005	2166	.....OO
ZZ4...	Open-hearth steel	B	0.047	0.389	0.040	0.027	0.007	0.033	1779	1285	2024	920	1991	1779	1630	......ZZ4
FF....	Open-hearth steel	A	0.280	0.500	0.027	0.038	0.010	0.034	1809	2328	3675	1779	3066	3097	2626	.....FF
BB....	Open-hearth steel	A	0.077	0.488	0.016	0.029	0.006	0.043	....	....	....	....	2823	2884*	2854	.....BB
MM....	Open-hearth steel	B	0.111	0.400	0.010	0.028	0.011	0.053	1470	2884	3341	2452	4136	2918	2867	....MM
BB....	Open-hearth steel	A	0.048	0.294	0.026	0.060	....	0.143	2729	3188	2085	3863	....	*	2966	.....BB
GG....	Open-hearth steel	A	0.290	0.510	0.028	0.039	0.010	0.217	3251	2976	3159	4136	4136	3341	3500	.....GG
TT....	Open-hearth steel	B	0.087	0.330	0.009	0.019	0.008	0.239	3675	3188	3830	3891	2519	2519	3270	.....TT
HH....	Open-hearth steel	A	0.069	0.371	0.010	0.026	0.004	0.248	2237	2298	3097	2793	2519	2729	2612	.....HH
ZZ5	Open-hearth steel	B	0.075	0.390	0.070	0.073	....	0.257	2394	1779	2852	2852	2452	2519	2475	......ZZ5
SS....	Open-hearth iron	B	0.015	0.021	0.006	0.020	0.005	0.023	951	3097	3251	3891	1409	2884	2581	......SS
DD....	Open-hearth iron	A	0.019	0.025	0.004	0.027	0.003	0.028	2729	3614	2729	3708	1193	1809	2630	.....DD
XX....	Open-hearth iron	B	0.028	0.060	0.009	0.028	0.006	0.030	2481	3647	982	3251	951	3863	2529	XX
CC ..	Open-hearth iron	A	0.020	0.027	0.005	0.033	0.003	0.078	4377	3769	3403	4227	1840	3097	3452	CC
CCC..	Open-hearth iron	A	0.018	0.028	0.005	0.032	0.006	0.262	2298	3675	2519	2976	3526	2085	2847	CCC
UU....	Open-hearth iron	B	0.014	0.075	0.006	0.028	0.007	0.250	3799	3830	3614	3923	4014	4136	3886	.....UU
VV	Open-hearth iron	B	0.009	0.050	0.008	0.018	....	0.288	2422	4136	4315	4377	2422	3675	3558	VV
AA....	Bessemer steel	A	0.030	0.420	0.085	0.040	0.003	0.012	1779	2729	1779	3066	3282	2519	2526	.....AA
ZZ....	Bessemer steel	B	0.080	0.440	0.080	0.032	....	0.014	951	2205	2143	2298	2519	2452	2095	......ZZ
KK....	Acid open-hearth steel	A	0.100	0.460	0.085	0.039	....	0.230	1779	1961	2607	2358	2452	2394	2259	.....KK
II....	Bessemer steel	A	0.060	0.360	0.091	0.066	0.005	0.254	1840	2237	2823	2519	2918	3037	2562	II
ZZ2...	Bessemer steel	B	0.078	0.340	0.091	0.036	....	0.287	1779	2143	2267	2085	1991	2328	2099	......ZZ2
YY....	Wrought iron	B	0.030	0.050	0.124	0.019	0.210	0.021	1409	2481	2607	2519	2669	2143	2505	.....YY
EE....	Wrought iron	B	0.033	0.026	0.111	0.019	0.109	0.307	2328	2519	3066	3495	3251	2519	2863	.....EE
					Average for each rack............				2205.4	2769.2	2856.5	3009.7	2575.7	2765.8		

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* Omitted from average for racks.

month. When the monthly inspection of all the racks in the box of No. 22 gage specimens was started, that did not affect the specimens in the box of No. 16 gage material. The latter were subjected to the same procedure only when the first failure in that gage occurred. Thus the No. 16 gage specimens were subjected to more continuous immersions of 6 months than were the No. 22 gage specimens.

This condition is to be especially noted because in analyzing the data, some may desire to give some weight to the relative lives of the No. 16 and the No. 22 gage specimens, the former being twice the thickness of the latter. Some may feel that the effect of thickness is an important factor. It is likely, however, that more happens to the specimens in six immersions of one month than happens in a continuous immersion of 6 months duration.

The figures given in Tables I to IV are the life of the specimens in elapsed time in days from the beginning of the test to the inspection at which failure was noted. The test was, however, an interrupted immersion test. The interruptions were made necessary by the method of inspection chosen by the members of Subcommittee V when the test was started.

Attempts to interpret the data and to come to some quantitative conclusions have been conscientiously made by the subcommittee. It must be apparent to anyone studying the results and considering the variables that have, during the life of the tests, expediently crept into the procedure, that there are too many factors of doubt to permit the drawing of more than very general conclusions as to which are the better materials of those tested, suitable for sea-water immersion without the benefit of some protective coating.

Conclusions:

The conclusions reached by the committee are submitted herewith for the consideration of the Society:

(*a*) The spread in time of failure is great.

(*b*) The difference between different kinds of material is small with respect to the spread in time.

(*c*) Because of these apparent factors the subcommittee decided at the time the last specimens of No. 22 gage material failed, that attempts to interpret the data would be made only after all the data were available.

(*d*) There is a difference between the different kinds of material at the two locations. The relative order of failure of the different materials at each of the two test locations is not the same.

(*e*) At Portsmouth, N. H. (both gages):

The three types of material, high-phosphorus steel, open-hearth iron, and basic open-hearth steel, in that order are consistent in their behavior in both gages. Considering the number of samples of wrought iron, the wrought iron has a lower life than the high-phosphorus steels. In the No. 16 gage open-hearth iron and the basic open-hearth steel and the wrought iron, the life has apparently been appreciably increased by the presence of copper. This is not true in the case of the No. 22 gage. Other elements produce little if any difference.

(*f*) At Key West, Fla. (both gages): The three types of material, high-phosphorus steel, open-hearth iron, and basic open-hearth steel are still consistent in their behavior in both gages but at this location the order is different from that at Portsmouth, and is as follows:

1. Open-hearth iron
2. Basic open-hearth steel
3. High-phosphorus steel

At this location the wrought iron, considering the number of samples, has about the same life. Here again the presence of copper has favorably affected the open-hearth iron, basic open-hearth steel, and the wrought iron, and has had little if any effect on the high-phosphorus steel in the No. 16 gage. In the No. 22 gage the effects of copper are less significant.

(*g*) With the exception of the high-phosphorus steel, the addition of copper has increased the life of all the materials under test at both locations.

(*h*) The open-hearth iron has lasted longer than the basic open-hearth steel in both the high- and low-copper groups.

Respectfully submitted on behalf of the subcommittee,

W. J. Jeffries,
Chairman.

REPORT OF SUBCOMMITTEE VIII ON FIELD TESTS OF METALLIC COATINGS

Report on Tests of Galvanized Sheets

Galvanized Coatings on Sheets at Sandy Hook:

Table I is an extension of tables shown in previous reports giving the record of coating failures at Sandy Hook, N. J. To date at this location, rust has been found on 104 out of the 108 specimens exposed there. This rack has been examined every year in April and October. Of the 104 specimens which show rust there, 80 (or 77 per cent of the total number rusted) have been discovered at the inspections made in October. It was postulated by some members of the subcommittee that the activity of the atmosphere in this place was greater between April and October than it was between October and April. This point was considered worthy of a supplemental investigation.

TABLE I.—RECORD OF COATING FAILURES ON SANDY HOOK, N. J., TEST RACK.
SHEETS EXPOSED MAY 20, 1926.

Description	Number of Specimens in Test	Up to Oct. 20,[a] 1937, 11.43 yr.	April, 1938, 11.94 yr.	Oct., 1938, 12.40 yr.	April, 1939, 12.94 yr.	Oct., 1939, 13.39 yr.	Total Number of Failures	Average Life of Coating in Years
No. 16 gage, 2.50 oz. per sq. ft.	12	6	none	4	none	2	12	11.79
No. 22 gage, 2.50 oz. per sq. ft.	18	7	1	2	2	2	14	(12.68)[b]
No. 22 gage, 2.00 oz. per sq. ft.	18	17	none	1	....	....	18	9.96
No. 22 gage, 1 50 oz. per sq. ft.	18	18	....	....	....	....	18	7.63
No. 22 gage, 1 25 oz. per sq. ft.	18	18	....	....	....	....	18	6.80
No. 22 gage, 0.75 oz. per sq. ft.	14	14	....	....	....	....	14	4.77
No. 28 gage, 0.75 oz. per sq. ft.	10	10	....	....	....	....	10	4.89
	108	....	1	7	2	4	....	....
Total failures to date	....	90	91	98	100	104	104	....

[a] A detailed record of the coating failures observed at the inspections up to October 20, 1937, inclusive, appears in Table III of the 1938 Report, *Proceedings*, Vol. 38, Part I, p. 86 (1938).

[b] Figures shown in parentheses are not to be considered final.

Accordingly, an experiment was started in 1939 in which samples of five 4 by 6-in. specimens of (*a*) sheet zinc and (*b*) galvanized steel sheet were exposed vertically and at 30 deg. from the horizontal. A sample representing each of these conditions was to be exposed for six-month periods from (*a*) April to October, 1939, (*b*) from October, 1939 to April, 1940, and (*c*) from April to October, 1940. Additional samples were to be exposed for twelve-month periods from April, 1939, to April, 1940, and from October, 1939, to October, 1940. The data obtained from the first inspection made in this supplemental investigation are shown in Table II. The specimens after exposure were cleaned free of corrosion products cathodically in a solution of disodium phosphate following which the specimens were washed, dried, and reweighed.

The loss of weight from the zinc sheet and from the galvanized steel sheet are not significantly different. Loss of zinc is being measured in both cases. The samples exposed at 30 deg. from the horizontal have lost more weight than those exposed vertically. From a qualitative examination of the pieces, the loss from the specimens exposed at 30 deg. was largely from the upper or exposed surface, whereas, the loss from the specimens exposed in the vertical position was divided about equally between the two surfaces. Evidence on the postulate which prompted this supplemental investigation is not, of course, contained in the data from the first inspection; that will come by comparison of these data with those obtained subsequently.

TABLE II.—LOSS OF WEIGHT IN GRAMS FROM SAMPLES OF FIVE 4 BY 6-IN. SPECIMENS EXPOSED 0.48 YR. AT SANDY HOOK, N. J., FROM APRIL 18, 1939 TO OCTOBER 11, 1939.[a]

	Exposed at 30 deg.		Exposed Vertically	
	Arithmetic Mean, $\overline{X}$	Standard Deviation, σ	Arithmetic Mean, $\overline{X}$	Standard Deviation, σ
Zinc sheet...	0.0606	0.0047	0.0324	0.0040
Galvanized steel sheet.	0.0614	0.0051	0.0314	0.0068
Average.....	0.0610	0.0049	0.0319	0.0054

[a] For method of presentation, see A.S.T.M. Manual on Presentation of Data, *Proceedings*, Vol. 33, Part I, p. 451 (1933). (Also available as separate publication.)

TABLE III.—RECORD OF COATING FAILURES ON STATE COLLEGE, PA., TEST RACK.[a]
SHEETS EXPOSED APRIL 23, 1926.

Description	Number of Specimens in Test	Up to Oct. 19, 1937,[a] 11.49 yr.	April, 1938, 12.02 yr.	April 23, 1939, 13.00 yr.	Oct. 6, 1939, 13.49 yr.	Total Number of Failures	Average Life of Coating in Years
No. 16 gage, 2.50 oz. per sq. ft...........	12	none	none	none	none	none	....
No. 22 gage, 2.50 oz. per sq. ft...........	18	none	none	none	none	none	....
No. 22 gage, 2.00 oz. per sq ft...........	18	none	none	none	none	none	....
No. 22 gage, 1.50 oz per sq. ft...........	18	none	none	none	none	none	....
No. 22 gage, 1 25 oz. per sq. ft...........	18	1	1	none	2	4	....
No. 22 gage, 0.75 oz. per sq. ft...........	14	14	....	....	....	14	10.04
No. 28 gage, 0.75 oz. per sq. ft...........	10	8	1	1	none	10	11.20
	108	....	2	1	2	....	....
Total failures to date....................	....	23	25	26	28	28	....

[a] A detailed record of the coating failures observed at the inspections up to October 19, 1937, inclusive, appears in Table IV of the 1938 Report, see *Proceedings*, Vol. 38, Part I, p. 87 (1938).

Galvanized Coatings on Sheets at State College:

Table III is an extension of similar tables shown in previous reports giving the record of coating failures at State College, Pa. It should be noted that at this locality the rusted sheets are still restricted to the lightest coating classes.

Information on the activity of other test locations (as judged by time required for the zinc to weather away sufficiently for rust to appear on the upper or exposed surface) has been given in previous reports similar to Tables I and III given here for Sandy Hook and State College. Table IV is included in this report to show briefly the relation of the five test locations in this respect.

TABLE IV.—TIME REQUIRED FOR APPEARANCE OF RUST ON NO. 22 GAGE SPECIMENS AT FIVE TEST LOCATIONS.

Location	Time, yr.[a]
Altoona, Pa.....................	1.89
Brunot Island (Pittsburgh), Pa....	2.18
Sandy Hook, N. J................	4.77
State College, Pa................	10.04
Key West, Fla..................	16.00[b]

[a] Average time required for first appearance of rust on upper surface of No. 22 gage specimen, 0.75 oz. per sq. ft. weight of coating class.

[b] Estimated from condition of upper surface after exposure of 13.84 yr. Actually, only 3 specimens out of 14 show any trace of such rust after that exposure.

Galvanized Sheets at Key West:

The information in Table IV indicates that at Key West, Fla., the coating on the upper surface of the sheets still remains largely intact. The behavior of the coating on the exposed surface is not of the same economic significance at Key West as at the other test locations.

Perforation of the sheets takes place at Key West from the under surface in the region around the lower purlin while the coating on the upper surface remains intact. So far as we are able to tell, water hangs on the under surface of the sheets around the lower purlin for relatively long periods of time. This water may orignate from dew (which is quite heavy on still nights in Key West), or from wind-borne spray or from driven rain. In any event, exposure to this sort of thing breaks down the coating on the under side. The base metal so exposed starts to rust from the under surface and the rusting proceeds through the sheet from the bottom surface to the upper surface. Figures 1 and 2 are illustrative of this condition.

TABLE V.—TIME IN YEARS AT WHICH THERE WAS FIRST INDICATION OF PERFORATION OF GALVANIZED SHEETS ON KEY WEST, FLA., TEST RACK.

SHEETS EXPOSED JUNE 9, 1926.

Description	Total	Jan. 29, 1937, 10.64 yrs.	March 22, 1938, 11.79 yrs.	March 21, 1939, 12.79 yrs.	April 10, 1940, 13.84 yrs.	Number of Failures	Average Life in Years
No. 16 gage, 2.50 oz. per sq. ft.	12	none	none	none	none	none	....
No. 22 gage, 2.50 oz. per sq. ft.	18	none	none	none	none	none	....
No. 22 gage, 2.00 oz. per sq. ft.	18	none	1	none	none	1	....
No. 22 gage, 1.50 oz. per sq. ft.	18	none	3	5	3	11	(14.7)[a]
No. 22 gage, 1.25 oz. per sq. ft.	18	2	4	4	2	12	(13.6)[a]
No. 22 gage, 0.75 oz. per sq. ft.	14	5	4	3	none	12	(12.1)[a]
No. 28 gage, 0.75 oz. per sq. ft.	10	2	3	2	1	8	(12.7)[a]
	108	9	15	14	6	....	....
Total failures to date	....	9	24	38	44	44	....

[a] Figures shown in parentheses are not to be considered final.

The first indication that corrosion has progressed through the sheet is seen as a spot of brown stain showing through the coating remaining on the upper surface. These spots so far have been found exclusively around the lower purlin. The appearance of these spots of brown stain does not mean that the sheets have served their entire usefulness as covering. This spot of brown stain is a very definite, readily observable point which precedes by some years the development of actual holes through the sheet. It is not subject to the misunderstanding that enters inspectors' minds over an actual hole through a sheet which may have been brought about by some mechanical damage or exaggeration by too close scrutiny and pecking. Table V is a record of the time in years at which this indication of perforation yet-to-come of the galvanized sheets on the Key West rack was observed.

Of the 44 specimens showing these brown-stained spots over the lower purlin, 26 (or 59 per cent) are on the east side of the rack. An examination of the under surfaces of the sheets on the two sides of the rack indicates the possibility that more damage is being done on the east side than on the west.

While the condition at Key West is unusual in comparison with the four northern locations, the developments there are thought typical of the deterioration observed in other tropical and semi-tropical places. The same sort of thing has been noted individually by members of the sheet inspection group on roofs in other parts of Florida, the Canal Zone and the Philippine Islands.

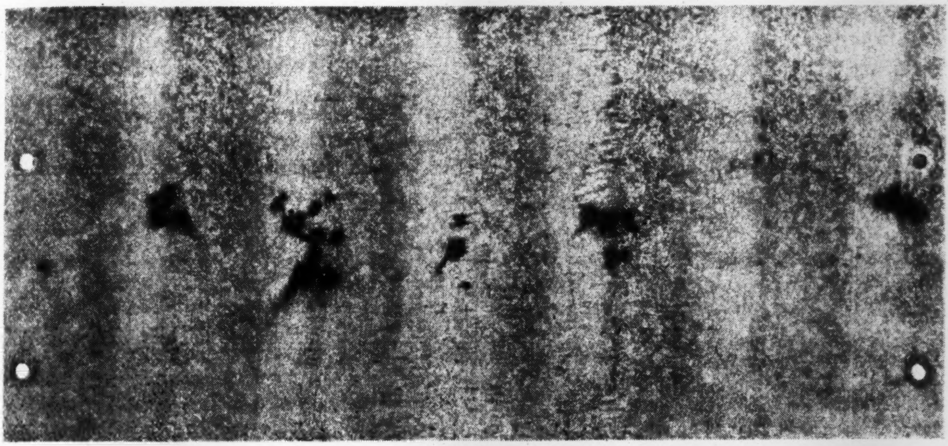

FIG. 1.—Typical Condition of Upper Surface of Sheet Showing Spots of Brown Stain.

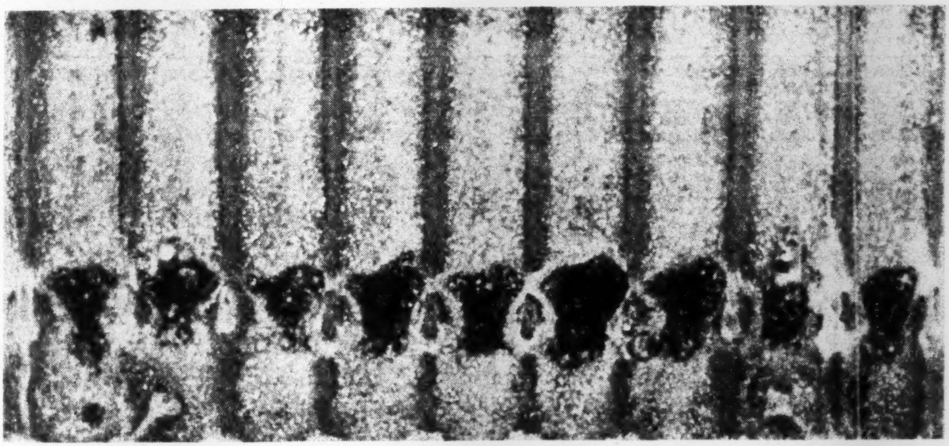

FIG. 2.—Under Surface of Same Sheet as Shown in Fig. 1.

So far as the present data go, it would appear that there is distinct value in the galvanized coating because its resistance to attack from the upper surface restricts damage of the base sheet to one surface. Although the coating fails on the under surface (too soon, some think), the protection given by the galvanized coating on the bottom surface apparently is affected by the weight of the coating.

In the 2.5-oz. coating class, no specimens yet show the brown stains. In the 2.0-oz. coating class, 5.6 per cent of the specimens now show the brown stains. In the 1.5-oz. coating class, 61.1 per cent of the specimens show brown stains. In the 1.25-oz. class, 66.6 per cent of the specimens show this condition. In the 0.75-oz. class, 85.7 per cent show this condition.

The test at Key West is still in progress and further observation certainly will clarify those points of interest on which the inspection committee can only conjecture now.

Uncoated Sheets:

At each of the five test locations, ten uncoated No. 22 gage sheets were exposed along with the galvanized sheets. These ten uncoated sheets represent the base-metal material appearing in the No. 22 gage galvanized sheets with several weights of coating. These uncoated sheet specimens have been inspected regularly for perforation according to the standards of Subcommittee III. Table VI shows the time in years which elapsed between exposure and the day on which perforation was found.

These uncoated sheets were included in the galvanized sheet test to show the contribution of the galvanized coating to the life of the galvanized sheets. When data on perforation of the galvanized sheets become available, these data on uncoated sheets will be essential to an understanding of the case. It is evident even now that the relative value of coating and base sheet depends on the location where the sheets are exposed.

TABLE VI.—TIME IN YEARS FOR PERFORATIONS OF NO. 22 GAGE, BLACK SHEETS.[a]

Identification	Class of Material	Average Chemical Composition									Brunot Island (Pittsburgh), Pa.	Altoona, Pa.	Key West, Fla.	Sandy Hook, N. J.	State College, Pa.
		Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Copper, per cent	Nickel, per cent	Chromium, per cent	Molybdenum, per cent					
R	Open-hearth iron...........	0.011	0.021	0.004	0.018	0.004	0.032	0.023	[d]	0.004[b]	2.03	4.27	3.62	5.94	....
V	Open-hearth iron...........	0.018	0.053	0.005	0.032	0.004	0.051	0.045	[d]	0.007[b]	2.27	4.27	3.62	4.93	....
U	Open-hearth iron	0.015	0.020	0.004	0.024	0.005	0.087	0.106	[d]	0.009[b]	2.53	4.27	3.62	5.42	....
N	Open-hearth iron...........	0.013	0.057	0.007	0.029	0.003	0.167	0.057	[d]	0.004[b]	3.00	7.51	3.62	5.94	....
T	Open-hearth iron...........	0.016	0.157	0.007	0.028	0.004	0.228	0.073	[d]	0.004[b]	9.00	8.52	3.62	6.45	....
Y	Open-hearth steel...........	0.028	0.346	0.067	0.050	0.005	0.029	0.003[b]	0.013	[c]	2.03	2.03	2.62	3.45	....
S	Open-hearth steel...........	0.040	0.488	0.072	0.041	0.003	0.051	0.006[b]	0.094	[c]	3.25	9.00	3.62	9.92	....
C	Open-hearth steel...........	0.066	0.458	0.053	0.045	0.003	0.222	0.003[b]	0.000[b]	[c]	12.48	X	5.73	10.42	....
K	Open-hearth steel...........	0.023	0.460	0.066	0.045	0.003	0.268	0.023[b]	0.031	[c]	....	13.47	4.58	12.39	....
W	Wrought iron...............	0.021	0.048	0.143	0.019	0.210	0.025	0.020[b]	[d]	0.003[b]	3.00	5.00	3.62	4.93	....

[a] Blanks in table represent sheets that have not failed as yet. The material has been under observation for approximately 13.5 yr.
[b] Average of determinations in one laboratory.
[c] Less than 0.003 per cent; results of one laboratory.
[d] Less than 0.006 per cent; results of one laboratory.
X Sheet lost due to breaking of straps.

Respectfully submitted on behalf of the subcommittee,

R. F. PASSANO,
Chairman.

REPORT OF COMMITTEE A-6
ON
MAGNETIC PROPERTIES

Committee A-6 on Magnetic Properties has held two meetings since its previous report: one in Atlantic City, N. J., on June 28, 1939, and one in Philadelphia, Pa., on March 25, 1940.

It is with deep regret that the resignation of W. J. Shackelton of the Bell Telephone Laboratories has been accepted. For many years he has made many valuable contributions to the work of this committee. J. A. Ashworth has been appointed to succeed Mr. Shackelton.

P. H. Dike of Leeds & Northrup Co. has been appointed chairman of Subcommittee I and as the representative of Committee A-6 on Committee E-8 on Nomenclature and Definitions, succeeding Mr. Shackelton. Mr. Ashworth has been appointed to Subcommittees I and IV of Committee A-6. Mr. Sanford has been appointed a member of Subcommittee III.

The present officers and members of the advisory committee were re-elected for the ensuing term of two years.

REVISION OF TENTATIVE STANDARD

The committee recommends that the Tentative Methods of Test for Measuring Interlamination Resistance and Lamination Factor of Iron and Steel (A 34 – 39 T)[1] be revised as indicated below and continued as tentative. The proposed revision affects the test for interlamination resistance. No change is being made in the procedure for lamination factor. No important criticisms of this particular method have been received but it is felt that a longer time than one year should be allowed for users to experiment with it and it is accordingly being continued as tentative.

Section 3.—Change the first sentence of Paragraph (*a*) to read as follows by the addition of the italicized figure and the omission of the words and figure in brackets:

> The test specimen shall consist of *40* small Epstein strips [weighing approximately 750 g., regardless of thickness,] 25 cm. ($9\frac{27}{32}$ in.) in length, and 3 ± 0.02 cm. ($1\frac{3}{16}$ ± 0.01 in.) in width, which shall be obtained from the Epstein core-loss test specimens.

Following this section add a note to read as follows:

> NOTE.—This method of test applies principally to the commercial electrical sheet thicknesses which are in the range of gages Nos. 24 to 29 (0.025 to 0.014 in.); for thicknesses outside this range, the number of strips and any other modifications of the test procedure, depending upon the application, shall be agreed upon by the manufacturer and the purchaser.

Section 4.—Add a new Paragraph (*c*) to read as follows, relettering the remaining paragraphs accordingly:

> (*c*) The specimen shall be subjected to a preloading pressure of 35.15 kg. per sq. cm. (500 psi.) before applying any test pressures.

In the present Paragraph (*d*) change the formula to read as follows by the addition of the italicized material and the omission of that in brackets:

$$R_s = \frac{AR}{S} = \left[\frac{75R}{S}\right] \mathit{1.875R}$$

To the explanation of the symbol "S" following the formula, add the figure "40."

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 990.

This recommendation has been submitted to letter ballot of the committee which consists of 25 members; 20 members returned their ballots, all of whom have voted affirmatively.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (P. H. Dike, chairman).—The only project before this subcommittee at present is a suggestion concerning the advisability of defining nonmagnetic steel and its properties. Perhaps a better term than "nonmagnetic" steel can be found for ferrous materials having permeabilities departing only slightly from one. There is also the question as to what should be considered the dividing line between nonmagnetic and ferromagnetic steels. Suggestions on these points would be welcome.

It is felt that the five definitions of incremental magnetic properties, accepted last year for publication as a tentative revision of the Standard Definitions of Terms, with Units and Symbols, Relating to Magnetic Testing (A 127 – 39), should be retained as tentative.

Subcommittee III on Direct Current Test Methods (B. M. Smith, chairman).—Two high coercive force magnet steel bars of the Alnico type have been checked by the laboratories of the General Electric Co. and the National Bureau of Standards in this country, the Metropolitan Vickers Co. and the National Physical Laboratory in England, and the Physikalisch-Technische Reichsanstalt and Allgemeine Elektrizitäts Gesellschaft in Germany. The residual induction values checked fairly well but there was some variation in the coercive force results as measured from a maximum magnetizing force of 2000 oersteds. These variations were from 410 to 380. The highest values were obtained by the General Electric Co. and the Metropolitan Vickers Co., and the lowest by the National Bureau of Standards and the A.E.G. These figures are not as close as had been hoped, which can probably be accounted for by radically different methods of test.

The General Electric Co. in collaboration with the National Bureau of Standards is now testing a link sample by the ring method which will then be cut up, giving two bars which will be checked in standard permeameters.

No standard method is available for the measurement of permeability of the so-called nonmagnetic steel materials, although various methods are known and in regular use. Subcommittee III will undertake the formulation of such test methods in the near future.

Subcommittee IV on Alternating Current Test Methods (J. P. Barton, chairman).—The method of measuring core losses and alternating-current permeability on small Epstein specimens has been established and specimens are now being tested by a number of laboratories to determine the reproducibility of the results. It is hoped that these tests will be completed in another year.

Subcommittee V on Magnetic Analysis (A. V. de Forest, chairman).—This subcommittee has been inactive during the past year and the desirability of discontinuing it is being considered.

This report has been submitted to letter ballot of the committee which consists of 25 members; 20 members returned their ballots, of whom 19 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

THOMAS SPOONER,
Chairman.

R. L. SANFORD,
Secretary.

REPORT OF COMMITTEE A-7

ON

MALLEABLE IRON CASTINGS

Committee A-7 on Malleable Iron Castings held two meetings during the year, one in Atlantic City, N. J., on June 26, 1939, at which 14 members and guests were present, and one in Detroit, Mich., on March 5, 1940, at which 17 members and guests were present.

The present membership of the committee now totals 36, of whom 16 are classified as producers, 11 as consumers, and 9 as general interest members; there are 2 consulting members.

During the past year the work of the committee and subcommittees has been chiefly of a routine nature. However, Subcommittee VI on Pearlitic and Alloy Malleable Iron (C. F. Lauenstein, chairman) has followed the suggestion made at the last annual meeting that further information be obtained on the physical properties of pearlitic malleable irons. It has been felt that the Tentative Specifications for Pearlitic Malleable Iron Castings (A 220 – 39 T) are too general and that they should cover more specific physical properties. Through the auspices of Subcommittee VI arrangements were made for C. H. Lorig of the Battelle Memorial Institute to submit a paper covering the physical properties of pearlitic malleable irons as they are manufactured today. This paper entitled "Properties of Commercial Pearlitic Malleable Irons"[1] is being presented at this annual meeting. It is possible that Mr. Lorig's paper will be the basis for further discussion and revision of Specifications A 220 – 39 T.

Another paper on the subject of malleable iron is also being presented at this annual meeting entitled "Stress-Strain Relations for Malleable Cast Iron in Tension with Special Attention to Yield Point Determination,"[2] by R. D. Landon of the Southern Methodist University.

Subcommittee VII on Welding had collected some information under the chairmanship of E. E. Griest, but because of Mr. Griest's resignation from the Society it was necessary to start anew the work of this subcommittee, and this has been undertaken under the chairmanship of W. E. Jones of the Stockham Pipe and Fittings Co. It is expected that a report of progress will be made by this subcommittee at the meeting of Committee A-7 to be held during this annual meeting.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, F. L. Wolf.
Vice-Chairman, J. J. Kanter.
Secretary, H. A. Schwartz.

This report has been submitted to letter ballot of the committee which consists of 36 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. K. Smith,
Chairman.

H. A. Schwartz,
Secretary.

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[1] Published in ASTM Bulletin, No. 105, August, 1940, p. 29.

[2] See p. 849.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held one meeting during the year: in Detroit, Mich., on March 5, 1940, during the spring group meetings of the Society.

Several changes in committee personnel have occurred during the year, including two resignations and the election of several new members and alternates. The present membership is 81, of whom 34 are classified as producers, 32 as consumers, and 15 as general interest members.

Subsequent to the 1939 annual meeting, Committee A-10 presented to the Society through Committee E-10 on Standards a Tentative Recommended Practice for Conducting Plant Corrosion Tests (A 224 - 39 T). The recommended practice was accepted[1] by Committee E-10 for publication as tentative on August 24, 1939, and appears in the 1939 Book of A.S.T.M. Standards.[2]

The preparation of specifications for bars, tubing, and wire has again been delayed or postponed for various reasons. Specifications for castings and strip products, adopted as standard last year, have not been altered; consideration is, however, being given to enlargement of the sheet specifications and improvement of the castings specifications, as noted in more detail later in this report.

Committee A-10 was recently informed of the death of Mr. J. J. Maher, alternate member of the committee representing the American Chain Co., Inc. At the June meeting of the committee, a resolution was adopted expressing sympathy and regret at Mr. Maher's untimely passing.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (E. F. Cone, chairman; Russell Franks, secretary).—The data being compiled by this subcommittee are rapidly being correlated and assembled for publication and will be referred to letter ballot of the committee shortly. The data include chemical analysis, mechanical properties, heat treatment, etc., of the various stainless steels and are to replace and bring up to date similar information published by the Society in 1930.[3] Nickel-base alloys are excluded from the present data, which cover the more important grades of stainless steels. The nature of the information to be included in the tables being prepared is such that every effort is being made to effect their publication by the Society as soon as possible.

Subcommittee IV on Methods of Corrosion Testing (E. S. Taylerson, chairman).—The special Subcommittee on

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee A-10 reported results of the letter ballot vote as follows: Of a total membership of 75, 48 members returned their ballots, of whom 43 voted affirmatively, 2 negatively, and 3 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 909.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, Plates V to XV (1930).

Inspection of Stainless Steel Architectural Structures is to be continued for five years with another inspection at the end of that period. The first report of this subcommittee was published last year.[4]

A supplementary report presenting the results of an examination of two formed sections of stainless steel removed from one of the buildings covered in the 1939 report is presented in the Appendix.

Tests on wire products under the jurisdiction of Committee A-5 on Corrosion of Iron and Steel are being followed and cooperation is being continued.

This subcommittee is responsible for the preparation of the Tentative Recommended Practice for Conducting Plant Corrosion Tests (A 224 – 39 T),[2] mentioned earlier in this report.

Further consideration is being given to a boiling liquid test procedure and general agreement of the members concerning this test should be reached shortly. The Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 – 39 T), developed by Committee B-3, has been reviewed and although favorably received it will be further discussed as to its applicability to materials under the jurisdiction of Committee A-10.

An extensive series of galvanic couple tests of stainless steels and other metals immersed in sea water and exposed to the atmosphere are being planned by Committee B-3 following preliminary work of this nature undertaken about two years ago, wherein stainless alloys were coupled with nickel, zinc, and copper. Liaison with this work is being established, C. C. Snyder having been selected to represent Committee A-10. Assistance will be given by Committee A-10 in selecting the proper types of stainless steel for test and following the actual tests. Atmospheric tests on stainless materials will not be considered by the committee in view of the various tests now being made by consumer and producer interests.

Subcommittee V on Mechanical Tests (V. Krivobok, chairman).—This subcommittee is continuing its study of the mechanical and elastic properties of stainless steels. Some progress has been made and results have been secured but they are not yet ready for publication.

Subcommittee VI on Metallography (Russell Franks, chairman).—In continuing its study of structural constituents and etching of the 18 per cent chromium, 8 per cent nickel types of alloy steel, the subcommittee will conduct tests for susceptibility to intergranular corrosion and will correlate the results obtained with photomicrographs already prepared.[5] This will be done on the original material if sufficient specimen material is available; otherwise new material will be selected and heat treated as before. Some duplication of microscopic work may be required.

Additional etching experiments using the cyanide and Murakami's etch tests to detect carbide are also proposed.

Subcommittee VII on Welding (L. C. Bibber, chairman).—The principal duty of this subcommittee in the welding of stainless steel is to serve as a liaison group to keep Committee A-10 advised of the activities of other committees interested in this subject. Subcommittee IV of the Engineering Research Foundation is a research committee interested in the welding of stainless steel, and has been the principal group with which Subcommittee VII has been keeping in close contact.

A meeting of Subcommittee VII was held in Detroit, Mich., in March, 1939, at which the relation between the different groups was discussed. At this meeting G. F. Jenks was desirous of knowing whether there was sufficient interest to justify the formation of a committee in the American Welding Society to codify welding practices used in the building of high alloy steel structures. Subsequently a meeting was called in New York, N. Y. and a committee was formed to be known as the Committee on Standards for Fusion Welding High Alloy Steel Engineering Structures. Subcommittee VII is represented on this committee as well as on Subcommittee IV of the Engineering Research Foundation, and will keep Committee A-10 advised of the activities of these various groups.

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 197 (1939).

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 203 (1939).

To further coordinate the work of Subcommittee VII with that of the American Welding Society and the Engineering Research Foundation on welding problems involved in stainless steel alloys, it has been proposed that the following be added to the personnel of Committee A-10:

L. H. Johnson, chairman of the newly organized American Welding Society Committee on Standards for Fusion Welding High Alloy Steel Engineering Structures, and

L. S. Hoyt, chairman of Committee IV on High Alloy Steels, Industrial Research Division, Welding Research Committee.

As intimated, codification of welding practices used in building high alloy steel structures will be undertaken by these groups together with corrosion testing of welds, with Subcommittee VII assisting and advising whenever possible.

Subcommittee VIII on Specifications for Wrought Products (F. B. Foley, chairman).—This subcommittee has been relatively inactive during the past year due to lack of demand for specifications for bars and forgings. Consideration is being given to the formulation of specifications covering wire products.

Subcommittee IX on Specifications for Flat Products (C. C. Snyder, chairman).—The following specifications for stainless steel flat products have been adopted as standard and the subcommittee has found no need during the year to revise or change them:

Standard Specifications for:

Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate (A 167 - 39),
Corrosion-Resisting Chromium Steel Sheet Strip, and Plate (A 176 - 39), and
High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 - 39).

Subcommittee IX is now engaged in the preparation of specifications covering grades of chromium-nickel alloy steels to be used in the construction of unfired pressure vessels to meet the needs of the A.S.M.E. Boiler Code Committee. A preliminary meeting to consider suggestions made by the Stainless Steel Technical Committee has already been held following which Subcommittee IX completed proposed Tentative Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels, which will be submitted to the Society subsequent to the annual meeting.[6]

Subcommittee X on Specifications for Castings (W. J. Jeffries, chairman).—This subcommittee has condensed its original ten specifications for alloy-steel castings into the following four standards:

Standard Specifications for:

20 per cent Chromium, 9 per cent Nickel Alloy-Steel Castings (A 198 - 39),
Chromium Alloy-Steel Castings (A 221 - 39),
Chromium-Nickel Alloy-Steel Castings (A 222 - 39), and
Nickel-Chromium Alloy-Steel Castings (A 223 - 39).

Information on the utility of these standards and need for revision is to be collected by means of a questionnaire to be directed to both consuming and producing interests. Following a study of the replies, the subcommittee plans to revise the existing standards, where necessary, to make them satisfactory to the greatest number of producers and consumers.

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[6] See Editorial Note, p. 117.

Subcommittee XI on Specifications for Tubular Products (Newell Hamilton, chairman).—Specifications for tubular materials for high-temperature service, including stainless steel grades, have been prepared by Committee A-1 on Steel; these cover alloy-steel pipe and alloy-steel boiler and superheater tubes. A recent request for specifications to cover corrosion-resistant steel tubing is receiving consideration.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 81 members; 42 members returned their ballots, of whom 40 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

EDITORIAL NOTE

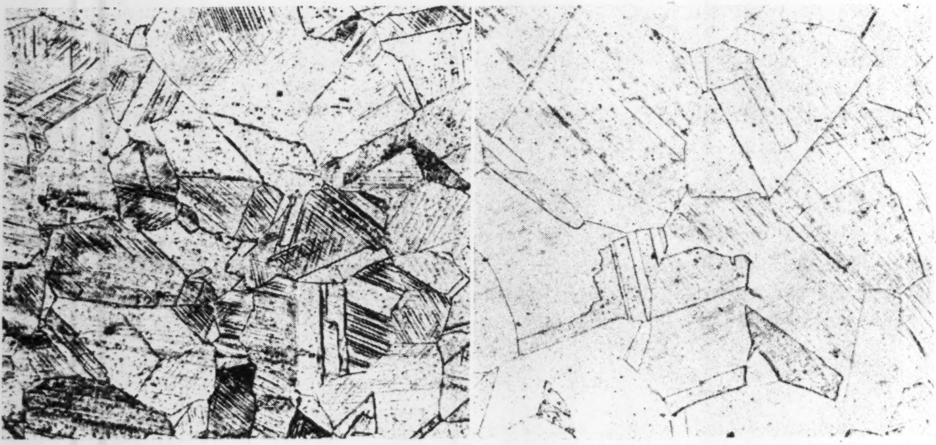
Subsequent to the annual meeting Committee A-10 presented to the Society through Committee E-10 on Standards new Tentative Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels. These specifications were accepted by Committee E-10 on August 28, 1940, and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 280, bearing the designation A 240 – 40 T.

APPENDIX

SUPPLEMENTARY REPORT ON INSPECTION OF STAINLESS STEEL ARCHITECTURAL STRUCTURES

An opportunity was provided during the year to examine two stainless steel formed sections which had been removed from the Chrysler Building in New York City. An inspection of the stainless steel on this building made on December 9, 1938, was discussed in the Report of Special Subcommittee on Inspection of Stainless Steel Architectural Structures.[1]

The two No. 24 gage formed sections which had been damaged by icing during the winter were removed from the gargoyles on the exterior of the building after having been in service for eleven years. An examination, including a chemical analysis and the preparation of photomicrographs of both samples, was made in the laboratory of the chairman of the special committee.



Sample No. 1.—Cold-worked austenitic structure.

Sample No. 2.—Coarse-grained austenitic structure.

FIG. 1.—Material Etched (Electolytic) in Chromic Acid (× 100).

The results of the chemical analysis of the stainless steel sections are as follows:

Sample	Carbon, per cent	Manganese, per cent	Silicon, per cent	Chromium, per cent	Nickel, per cent
No. 1	0.07	0.45	0.055	17.50	9.30
No. 2	0.07	0.45	0.056	17.40	9.20

The results of the chemical analyses indicate that both sections were 18 per cent chromium, 8 per cent nickel alloy steel. The close similarity of the analyses on both samples indicates they were from the same heat of steel. Microexamination revealed a relatively coarse-grained structure on both samples as shown by the photomicrographs in Fig. 1. Numerous cold-worked areas which probably resulted from forming during fabrication were present on both sections. Both sections, except for mechanical damage which had resulted from icing during the winter, were in excellent condition. There was no evidence of corrosive attack on any part of either section.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 197 (1939).

Respectfully submitted on behalf of the special subcommittee,

C. C. SNYDER,
Chairman.

REPORT OF THE RESEARCH COMMITTEE

ON

FATIGUE OF METALS

During the year the Research Committee on Fatigue of Metals held a meeting at Detroit, Mich., on March 5, 1940, during the spring meetings of A.S.T.M. committees. At this meeting the progress of the investigation of the effect of type of testing machine on results of fatigue tests was discussed, and plans laid for its further progress. Size effect was shown in the tests, and some evidence obtained indicating that the shape of cross-section of the specimen modified the endurance limit.

In carrying on the investigation of the effect of type of testing machine on fatigue test results, nine laboratories have been cooperating with the committee as follows:

U. S. Naval Aircraft Factory, Philadelphia, Pa.,
U. S. Air Corps Laboratories, Wright Field, Dayton, Ohio,
National Bureau of Standards, Washington, D. C.,
Westinghouse Research Laboratories, East Pittsburgh, Pa.,
U. S. Naval Engineering Experiment Station, Annapolis, Md.,
University of Illinois,
Massachusetts Inst. of Technology,
American Steel Foundries, East Chicago, Ind., and
Aluminum Research Laboratories, New Kensington, Pa.

The Timken Roller Bearing Co., Canton, Ohio, and the American Rolling Mill Co., Middletown, Ohio, have furnished the committee with specially prepared lots of steel for the investigation.

An oral presentation of the results to date will be given at the 1940 annual meeting of the Society.

The cooperation of the committee with *Metals and Alloys* in preparing and editing abstracts of articles on fatigue of metals has been continued.

Respectfully submitted on behalf of the committee,

H. F. Moore,
Chairman.

REPORT OF JOINT RESEARCH COMMITTEE
ON
EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

Progress Report to the Sponsor Societies

Meetings:

During the past year, the Joint Research Committee on Effect of Temperature on the Properties of Metals under the sponsorship of the American Society of Mechanical Engineers and the A.S.-T.M., held one meeting: on November 3, 1939, in Columbus, Ohio. A second meeting of the committee will be held in June, 1940, in Atlantic City, N. J., prior to the presentation of this report at the annual meeting of the A.S.T.M. A number of meetings of project committees have been held during the year.

Finances:

There appears in Table I a summary of the administration of funds during the past year, as of April 30, 1940.

Expenditures have been authorized and payments made to date on several research projects, as follows:

	Maximum Expenditure Authorized	Payment Made
Project No. 10—Tubular Members Subjected to Internal Pressures..	$3500	$1400
Project No. 13—Properties of Metals at Low Temperatures.......	500	None
Project No. 18—Effect of Variables on the High-Temperature Properties of Metals (Part 3).........	4000	None

Since the last progress report to the sponsors, Dean C. E. MacQuigg has been serving as chairman of the Finance Committee. Under Dean MacQuigg's direction the committee is now engaged in soliciting funds from industry for the continuation of its research program. To date the results of this solicitation are encouraging to the membership, indicating the continued interest and willingness to support the type of work being carried out by the committee. The committee hopes that further financial support will be forthcoming during the present year in order that other important research activities may be instituted.

Organization and Membership:

There have been no changes in the membership of the Joint Research Committee during the past year. Aside from the appointment of Dean C. E. MacQuigg as chairman of the Finance Committee, and of H. J. Kerr as the Chairman of Project No. 10, and the formation of Project Committees Nos. 24, 25, and 26, there have been no changes in the organization of the committee.

The present officers (N. L. Mochel, chairman; H. J. Kerr, vice-chairman; J. W. Bolton, secretary; and R. H. Aborn and H. J. French, members-at-large of the executive committee) have been re-elected to serve for another period of two years.

Research Projects:

There follows a summary of progress made in the various research projects during the year ending June, 1940.

Project No. 10 on Tubular Members Subjected to Internal Pressures (H. J. Kerr, chairman).—This project is a continuation of work previously reported,[1] using the same general methods and equipment, the work being carried out at the Massachusetts Institute of Technology by F. H. Norton. The project includes seven creep tests on tubular specimens subjected to internal pressures and four tension creep tests, on carbon-molybdenum steel, at temperatures of 900 and 1050 F. The estimated cost is $3500, a grant of $2000 having been secured from the Engineering Foundation, the remaining $1500 being from funds provided by industry. The project is well under way at the present time.

TABLE I.—SUMMARY OF RECEIPTS AND EXPENDITURES OF COMMITTEE FUNDS.

Balance as of May 31, 1939, shown in 1939 report				$3324.71
	RECEIPTS			
1939				
September	Interest on funds		$71.82	
	Refunds from University of Michigan:			
	From Project No. 15	$69.47		
	From Project No. 17	16.99	86.46	
November	The Engineering Foundation		500.00	
1940				
January	The Engineering Foundation		500.00	
	1939-40 Solicitation for funds		3800.00	
February	1939-40 Solicitation for funds		3400.00	
March	1939-40 Solicitation for funds		400.00	
April	A.S.T.M.-Refund on Project No. 11		312.00	
	The Engineering Foundation		500.00	
	Climax Molybdenum Co.—For Project No. 25		1000.00	
	1939-40 Solicitation for funds		550.00	
	Total Receipts			11 120.28
				14 444.99
	EXPENDITURES			
1939				
June	Battelle Memorial Institute (Project No. 18)		$414.40	
July	Battelle Memorial Institute (Project No. 18)		857.74	
	Duplicating First Draft of Project No. 20 Report		35.00	
August	Battelle Memorial Institute (Project No. 18)		144.79	
September	Battelle Memorial Institute (Project No. 18)		99.24	
October	Battelle Memorial Institute (Project No. 18)		275.35	
	Ogilvie Press (letterheads)		1.60	
	Mack Printing Co. (reprints)		6.90	
November	Battelle Memorial Institute (Project No. 18)		224.71	
December	Battelle Memorial Institute (Project No. 18)		195.86	
1940				
January	Battelle Memorial Institute (Project No. 18)		213.24	
February	Battelle Memorial Institute (Project No. 18)		61.65	
April	Battelle Memorial Institute (Project No. 18)		124.16	
	Massachusetts Inst. of Technology (Project No. 10)		1400.00	
	Expense of Finance Committee		43.86	
	Battelle Memorial Institute (Project No. 18)		186.72	
	Mack Printing Co. (reprints)		10 00	
	Total Expenditures			4295.22
	Balance on hand, April 30, 1940			$10 149.77

Project No. 13 on Properties of Metals at Low Temperatures (F. B. Foley, chairman).—Mr. Foley prepared a report[2] on the Round-Table Discussion of Effect of Subatmospheric Temperatures on the Properties of Metals held at Atlantic City, N. J., on June 27, 1939.

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[1] F. H. Norton, "Creep in Tubular Pressure Vessels," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 61, pp. 239 to 245, April, 1939.

The project committee has prepared special forms to facilitate the gathering of data on the properties of metals at low temperatures, and these will be distributed to laboratories and industrial concerns during the coming year.

Project No. 16 on Relaxation (E. L. Robinson, chairman).—In response to a growing need for reliable information on bolting performance at high temperature, Project No. 16 has offered to sponsor a session on this subject. Such a session will be organized if and when a suitable volume of data is in the hands of the subcommittee or offered for presentation. The holding of such a session and the publication of its proceedings would conclude all the activities at present contemplated for Project No. 16.

Project No. 18 on Effect of Variables on the High-Temperature Properties of Metals (H. W. Gillett, chairman).—In Appendix V to its 1938 report,[3] the committee presented the first progress report on this most important consideration. During the past two years, this research has been continued and a second progress report appears as Appendix I to this report.

The committee has arranged to continue this investigation and has authorized a further expenditure of $4000 to cover the work.

The importance of the results of these investigations to all organizations engaged in the manufacture and application of materials, or the preparation of specifications for materials for high-temperature service is apparent to all interested in the subject.

Project No. 20 on Grain Size (R. H. Aborn, chairman).—This project committee prepared an introduction to the subject of the grain size of steel, which has been published by one of the sponsors.[4] The committee believes that this presentation will be of value to engineers, especially to those who meet with and must consider the effect of grain size and structure on the high-temperature properties of steels.

Project No. 21 on Listing of High-Temperature Testing Equipment and Methods (G. F. Jenks, chairman).—This project committee has been in touch with all laboratories in the country known to be engaged in high-temperature testing. Data from 54 laboratories have been received and are now being indexed and assembled.

Project No. 22 on Correlation of Data on K-20 Steel (P. E. McKinney, chairman).—Since the preparation of this lot of 0.35 per cent carbon steel in 1935, wide use has been made of it in high-temperature testing. Probably no other single lot of material has been tested in so many ways and in so many laboratories, and the results are useful, therefore, in comparing or correlating test methods, laboratories, and in many other ways. The committee felt that it would serve a useful purpose to prepare a list of the various tests made on this material. This list, prepared by Project Committee No. 20, appears as Appendix II to this report.

Project No. 23 on Non-Ferrous Metals and Alloys (A. B. Bagsar, chairman).—A considerable number of non-ferrous alloys are being used in various industries at temperatures up to 1000 F.

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[2] F. B. Foley, "Résumé of Round-Table Discussion on Effect of Subatmospheric Temperatures on the Properties of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 637 (1939).

Cyril Stanley Smith, "Mechanical Properties of Copper and Its Alloys at Low Temperatures: a Review," *Ibid.*, p. 642.

H. Habart and W. J. Herge, "Sub-Size Charpy Relationships at Sub-Zero Temperatures," *Ibid.*, p. 649.

Walter Crafts and John J. Egan, "Factors Affecting Notched-Bar Impact Tests on Steel at Low Temperatures," *Ibid.*, p. 659.

[3] Howard C. Cross and J. G. Lowther, "Progress Report on Study of Effects of Manufacturing Variables on the Creep Resistance of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 149 (1938).

[4] J. R. Vilella, "The Grain Size of Steel," *Mechanical Engineering*, April, 1940, p. 293.

However, no concordant data are available in the literature on the high-temperature properties of these alloys and no correlated tests are now in progress in laboratories to obtain such data. Plans are being worked out for conducting exploratory tests on a cooperative basis for studying some of the important properties of copper-base and nickel-base alloys at high temperatures.

Project No. 24 on Influence of Time, Temperature and Stress on the Properties of 0.50 per cent Molybdenum Steels (A. J. Herzig, chairman).—A project is in course of planning that will attempt to determine the boundaries of the field in which carbon-molybdenum steel may be subject to premature failure.

Project No. 25 on Comparison of Short-Time Test Methods (P. E. McKinney, chairman).—A project is in course of planning that will compare the results of a number of so-called "short-time" test methods, and attempt to evaluate these methods in terms of their ability to forecast long-time (creep) test results. Several of the European short-time creep test methods will be included.

Project No. 26 on Test Methods (J. W. Bolton, chairman).—The committee is recommending that the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 37 T)[5] and the Tentative Method of Test for Long-Time (Creep) High - Temperature Tension Tests of Metallic Materials (E 22 – 38 T)[5] be continued as tentative for another year. A project committee has been appointed to study these test methods critically, with their adoption as standard in view. The project committee will also consider the possible need for preparing other test methods.

This report has been submitted to letter ballot of the committee which consists of 22 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the Joint Research Committee,

N. L. MOCHEL,
Chairman.
H. J. KERR,
Vice-Chairman.

J. W. BOLTON,
Secretary.

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[5] 1939 Book of A.S.T.M. Standards, Part I, pp. 1254 and 1261, respectively.

APPENDIX I

STUDY OF EFFECTS OF VARIABLES ON THE CREEP RESISTANCE OF STEELS

(PROJECT NO. 18)

PREPARED BY H. C. CROSS AND J. G. LOWTHER

SECOND REPORT COVERING A SPONSORED RESEARCH PROJECT CARRIED OUT AT BATTELLE MEMORIAL INSTITUTE

SYNOPSIS

This progress report shows results of creep tests at 850 F. on two pure irons, one with 0.5 per cent silicon added; on steel from rimmed and silicon-killed ingots from the same heat (0.06 to 0.08 per cent carbon), and on a rimmed steel (0.16 to 0.26 per cent carbon) and four different killed steels with 0.14 to 0.17 per cent carbon.

All materials were heat treated to produce similar fine and coarse-grained structures for comparison of their relative creep resistance and to determine the effect of different deoxidation practices.

Heat treatment producing coarsening of a practically carbon-free pure iron and a pure iron containing 0.5 per cent silicon improved the creep resistance. Silicon-killed steel showed consistently better creep resistance than rimmed steel as heat treated to produce both fine- and coarse-grained material. In S.A.E. 1015 steels, silicon-killed steels are superior to silicon-aluminum and aluminum-killed steels as fine grained or as slow cooled from the coarsening treatment used, while as air-cooled from the coarsening temperature the steels showed similar creep resistance.

All steels showed improvement in creep resistance when air cooled from temperatures producing coarse-grained material as compared with air cooled from temperatures to produce fine-grained material with the greatest improvement resulting in the aluminum-treated steels which were considerably poorer than the silicon-killed steels as fine grained.

INTRODUCTION

A previous progress report to the sponsor societies[1] indicated decided superiority in creep resistance at 750, 850, and 950 F. for carbon steels from 0.27 to 0.58 per cent carbon with coarse austenite grain as compared with the same steels with fine austenite grain.

Steels of lower carbon content (0.10 to 0.20 per cent) are also used for high-temperature service so a study of the effect of austenite grain size on their creep resistance was a logical step in the broad study of the variables affecting creep resistance. In fact, the question was raised as to whether the marked effect of austenite grain size on creep resistance would be present, less potent, or absent in carbonless pure iron, or whether the effect would be obtained only in steels containing appreciable amounts of carbon.

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[1] H. C. Cross and J. G. Lowther, "Progress Report on Study of Effects of Manufacturing Variables on the Creep Resistance of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 149 (1938).

Since the previous progress report indicated deoxidation practice and grain size as major variables in creep resistance, any study of lower carbon steels (0.10 to 0.20 per cent) should include silicon-killed, silicon-aluminum-killed, and aluminum-killed steels of otherwise similar chemical composition.

Previous data[2] have indicated rimmed steel to be inferior in creep resistance to killed steel. The effect of variations in grain size were not studied in those experiments. Since different deoxidation and finishing practices are used in the production of rimmed and killed steels, different grain-coarsening characteristics are sure to be present. From both a fundamental and a practical standpoint it is necessary to determine whether the poorer creep resistance of rimmed steel is due to a finer austenite grain or whether the poorer properties result from some effect of the steel finishing practice not indicated by austenite grain size.

Deep etching of rimmed steel billets shows well-defined skin and core areas differing in appearance as etched. These are due to progressive solidification and resulting segregation of rimmed steel. In the low carbon ranges (0.06 per cent carbon), the carbon content is quite uniform but grain-coarsening characteristics of the core and skin are different. In the high carbon ranges (0.15 to 0.20 per cent carbon), marked carbon segregation also occurs. Determination of the creep resistance of the skin and core material heat treated to comparable grain sizes would give information as to the variation in creep resistance across sections of rimmed steel.

Irons and steels of appropriate analysis and methods of finishing and deoxidation have been obtained from several sources. This report presents the test data obtained during their study.

STEELS TESTED

The steels studied are listed in Table I along with their chemical composition and some details of their deoxidation practice.

Through the courtesy of N. L. Mochel, T. D. Yensen, and the Westinghouse Electric and Manufacturing Co., very pure iron specimens were supplied. Pure iron 371-9 was obtained from a 50-lb. ingot made by melting electrolytic iron under dry hydrogen. It had a carbon content of only 0.003 per cent. This pure iron was obtained so as to determine whether carbonless iron would show effects of austenite grain size on creep resistance similar to those shown by steels with appreciable amounts of carbon. Iron 371-8 was obtained by adding 0.5 per cent silicon to electrolytic iron deoxidized with carbon and decarburized with moist hydrogen. Iron 371-8 allows a study of the effect of silicon on the creep resistance of carbonless iron heat treated to different grain sizes. Neither of the irons shows any carbides present, but the irons are not entirely free from oxide inclusions.

Through the courtesy of J. H. Nead and the Inland Steel Co., 4 by 4-in. billets were supplied from rimmed and silicon-killed ingots taken from the same heat. Full details of the furnace practice were supplied. The ladle addition for the whole heat was 80 per cent ferromanganese and $\frac{1}{4}$ lb. per ton of aluminum. Ingot No. 4 supplied the billet of rimmed steel. In the 22 by

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[2] A. E. White and C. L. Clark, "Creep Characteristics of Metals at Elevated Temperatures," *Transactions*, Am. Soc. Steel Treating, Vol. 21, pp. 1-22 (1933).

24 by 72-in. mold used, 3 oz. of aluminum were used to control the rimming action which was normal. Ingot No. 5 supplied the billet of silicon-killed steel. Twelve pounds of 84 per cent ferrosilicon were added uniformly as the ingot (22 by 24 by 72-in.) was poured. This killed the steel effectively and the ingot solidified with a slightly bulged top. The carbon content of the rimmed steel was 0.06 per cent, and of the silicon-killed steel 0.08 per cent. The silicon-killed steel contained 0.085 per cent silicon. The analyses showed very little difference in composition between the core and skin of the silicon-killed billet. Deep etching of sections of the rimmed and silicon-killed billets showed a definite core only in the rimmed billet. Material from both the core and the skin of the rimmed steel and silicon steel was selected for test.

TABLE I.—CHEMICAL COMPOSITION OF THE STEELS TESTED.

Steel Identification and Deoxidation Practice	Chemical Composition, per cent					
	Carbon	Manganese	Silicon	Sulfur	Phosphorus	Aluminum Addition
Pure iron—371-9	0.003					
0.5 per cent silicon iron 371-8	0.003		0.5			
Rimmed-core	0.06	0.36	nil	0.045	*a*	0.012^{b}
Rimmed-skin	0.06	0.41	nil	0.023	*a*	0.012^{b}
Silicon-killed-core	0.08	0.39	0.084^{c}	0.031	*a*	0.012
Silicon-killed-skin	0.08	0.41	0.086^{c}	0.031	*a*	0.012
Rimmed-core	0.26	0.51	nil	0.039	0.009	*d*
Rimmed-skin	0.16	0.46	nil	0.021	0.008	*d*
5247-silicon-killed	0.14	0.48	0.18^{e}	0.03	0.014	
3194-silicon-aluminum-killed	0.15	0.47	0.16^{f}	0.025	0.017	*g*
5229-aluminum-killed	0.15	0.45	0.02	0.020	0.011	*h*
5280-silicon-aluminum-killed	0.17	0.48	0.16^{i}	0.030	0.020	*i*

[a] Ladle, 0.008 per cent phosphorus.
[b] 3 oz. aluminum used to control rimming action in 22 by 24 by 72-in. ingot in addition.
[c] 12 lb. of 84 per cent ferrosilicon added uniformly as the ingot (22 by 24 by 72-in.) was poured.
[d] ½ lb. per ton of ferrotitanium added to ladle.
[e] 10 lb. of 50 per cent ferrosilicon added per ton as ladle addition.
[f] 7.6 lb. of 50 per cent ferrosilicon added per ton as ladle addition.
[g] 1 lb. aluminum per gross ton added as ladle addition.
[h] 2 lb. aluminum per gross ton added as ladle addition.
[i] Heat finished with 0.16 per cent silicon plus 2 lb. per ton of aluminum to produce fine grain.

The Inland Steel Co. also supplied a 4 by 4-in. billet from a rimmed ingot with a higher carbon content than 0.06 per cent. The ladle addition for this heat was ferromanganese and ferrotitanium (½ lb. per ton). As was expected, the ingot showed marked carbon segregation, analyzing 0.12 per cent carbon at the bottom, 0.16 per cent carbon at the middle, and 0.16 per cent (skin) to 0.26 per cent (core) at the top of the ingot. Use of the top portion of the ingot permitted a study of the effect of the segregation in this rimmed-steel billet since test material was taken from both the skin and core.

Through the courtesy of R. H. Aborn, and E. C. Wright, and the National Tube Co., 1-in. round bar stock was supplied from four S.A.E. 1015 heats of similar composition with 0.14 to 0.17 per cent carbon. Heat 5247 was silicon killed and had 10 lb. of 50 per cent ferrosilicon per ton as a ladle addition. Heat 3194 was silicon-aluminum killed (7.6 lb. of 50 per cent ferrosilicon per ton and 1 lb. of aluminum per ton were added). Heat 5229 was completely aluminum killed with an addition of 2 lb. of aluminum per ton. Heat 5280 was silicon-aluminum killed (7.6 lb. of 50 per cent ferrosilicon per ton) and 2 lb. of aluminum per ton were added.

These four heats from the National Tube Co. were melted similarly except for the final additions and therefore

TABLE II.—CREEP TEST DATA FOR TESTS AT 850 F. ON PURE IRON AND LOW-CARBON STEELS WITH VARIOUS OXIDATION PRACTICES AND HEAT TREATMENTS TO PRODUCE FINE AND COARSE AUSTENITE AND FERRITE GRAIN SIZES.

Steel Designation and Specimen Number	Carbon, per cent	Deoxidation Practice	Location of Test Material	Load, psi.	Average Initial Deformation, per cent	Rate of Deformation at 500 hr., per cent per hr.	Average Total Deformation at 500 hr., per cent	Heat Treatment	Austenite Grain Size, A.S.T.M. Number	Ferrite Grain Size
371-9-1	0.003[a]		⅞-in. rounds	6 000	0.075	0.007	3.096[i]	1292 F., 15 min. in H$_2$, air cool	5 to 8	fine
371-9-2A	0.003[a]		⅞-in. rounds	6 000	0.030	0.000085	0.105	1832 F., 10 hr. in H$_2$, air cool	0 to 2	[d]
371-9-2B2	0.003[a]		⅞-in. rounds	6 000	0.044	0.00073	0.460	1832 F., 10 hr. in H$_2$, then[b]	0 to 2	[d]
371-8-1	0.003[c]		⅞-in. rounds	6 000	0.073	0.00276	2.075	1292 F., 15 min. in H$_2$. air cool	[d]	[d]
371-8-2A	0.003[c]		⅞-in. rounds	6 000	0.034	0.000015	0.05	1832 F., 10 hr. in H$_2$, air cool	0 or larger	[d]
371-8-2B	0.003[c]		⅞-in. rounds	6 000	0.028	0.000336	0.156	1832 F., 10 hr. in H$_2$, then[b]	0 or larger	[d]
RI-5	0.06	rimmed	core	10 000	0.078	0.000854	0.890	1650 F., 2 hr., air cool	5 to 7	fine
RI-1-1	0.06	rimmed	core	10 000	0.064	0.00062	0.603	2000 F., 2 hr., air cool	2 to 4	fine
RI-8	0.06	rimmed	skin	10 000	0.095	0.0023	2.073	1650 F., 2 hr., air cool	5 to 7	fine
RI-4-1	0.06	rimmed	skin	10 000	0.092	0.0020	1.885	1825 F., 2 hr., air cool	2-4-some 5	fine
SI-1-1	0.08	Si-killed	core	10 000	0.068	0.000225	0.342	1625 F., 5 min., air cool	5 to 6 some 7	fine
SI-1-3	0.08	Si-killed	core	10 000	0.057	0.00016	0.276	1850 F., 2 hr., air cool	2 to 4	fine
SI-14	0.08	Si-killed	skin	10 000	0.063	0.00034	0.456	1625 F., 5 min., air cool	5 to 8 some 4	fine
SI-4-1	0.08	Si-killed	skin	10 000	0.059	0.00016	0.270	1750 F., 3 hr., air cool	2 to 4 some 1 & 5	fine
9-B	0.26	rimmed	core	15 000	0.119	0.0010	1.1	1560 F., 5 min., air cool	5 to 7	fine
8-B	0.26	rimmed	core	15 000	0.088	0.00078	0.727	2000 F., 1 hr., air cool	2 to 4	fine
12-C	0.16	rimmed	skin	15 000	0.330	0.00472[e]	2.86[e]	1560 F., 2 min., air cool	5 to 7	fine
3-B	0.16	rimmed	skin	15 000	0.187	0.00416	3.446	1950 F., 1 hr., air cool	2 to 4	fine
5247-19	0.14	Si-killed	1-in. rounds	15 000	0.112	0.000129	0.380	1575 F., 5 min., air cool	5 to 7 some 4	fine
5247-6	0.14	Si-killed	1-in. rounds	10 000	0.042	0.000015	0.082	1800 F., 1½ hr., air cool	2 to 4	fine
5247-6	0.14	Si-killed	1-in. rounds	15 000[f]	0.130	0.000045	0.223	1800 F., 1½ hr., air cool	2 to 4	fine
5247-16	0.14	Si-killed	1-in. rounds	15 000	0.212	0.000094	0.501	1800 F., 1½ hr., then[g]	2 to 4	coarse
3194-19	0.15	Si-Al killed[k]	1-in. rounds	15 000	0.119	0.000126	0.394	1575 F., 5 min., air cool	5 to 7	fine
3194-6	0.15	Si-Al killed	1-in. rounds	10 000	0.052	0.000015	0.091	1900 F., 2 hr., air cool	2 to 4	fine
3194-6	0.15	Si-Al killed	1-in. rounds	15 000[f]	0.127	0.000035	0.235	1900 F., 2 hr., air cool	2 to 4	fine
3194-16	0.15	Si-Al killed	1-in. rounds	15 000	0.273	0.000082	0.503	1900 F., 2 hr., then[g]	2 to 4	coarse
5229-19	0.15	Al-killed[l]	1-in. rounds	15 000	0.043	0.014	3.292	1600 F., 10 min., air cool	6 to 8 mostly 7	fine
5229-6	0.15	Al-killed	1-in. rounds	10 000	0.045	0.00001	0.073	1950 F., 2 hr., air cool	2 to 4	fine
5229-6	0.15	Al-killed	1-in. rounds	15 000[f]	0.117	0.00006	0.233	1950 F., 2 hr., air cool	2 to 4	fine
5229-16	0.15	Al-killed	1 in. rounds	15 000	0.378	0.000884	1.630	1950 F., 2 hr., then[g]	2 to 4	coarse
5280-9	0.17	Si-Al killed[e]	1-in. rounds	15 000	0.161	0.015[j]	2.767[j]	1730 F., 1 hr., air cool	7 to 8	fine
5280-1	0.17	Si-Al killed[e]	1-in. rounds	15 000	0.092	0.00006	0.165	2000 F., 1 hr., air cool	80% 2 to 4 some 1 & 5	fine
5280-6	0.17	Si-Al killed[e]	1-in. rounds	15 000	0.320	0.016[h]	2.935[h]	2000 F., 1 hr., then [g]	80% 2 to 4 some 1 & 5	coarse

[a] Pure iron with 0.003 per cent carbon.

[b] Furnace cooled from 1832 F. to 1560 F., held at 1560 F. for 10 hr., then the furnace cooled.

[c] Pure iron with 0 003 per cent carbon and 0.5 per cent silicon added.

[d] See text for discussion of structures obtained

[e] At 312 hr.

[f] At 550 hr. load raised from 10,000 to 15,000 psi.

[g] Furnace cooled from maximum temperature to 1575 F., then cooled 40 F. per hour to 1250 F., then furnace cooled.

[h] At 102 hr. test discontinued. [i] At 219 hr. [j] At 122 hr. [k] 1 lb. of aluminum per ton added. [l] 2 lb. of aluminum per ton added.

provide an excellent means of comparing silicon- and aluminum-killed steels, and silicon-aluminum-killed steels to which varying amounts of aluminum were added. These four killed heats (0.14 to 0.17 per cent carbon) can also be compared with the skin material of the rimmed heat which contained 0.16 per cent carbon.

CREEP TEST EQUIPMENT AND PROCEDURE

Descriptions of the creep test equipment and procedure at Battelle Memorial Institute have been given previously, especially in reports to the sponsor societies of the joint committee and will not be repeated here.[3, 4]

Throughout the series of creep tests herein reported the requirements of the A.S.T.M. Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 38 T)[5] have been met or exceeded.

As in the previous work reported 2 yr. ago,[1] 500-hr. creep tests were used to show extreme differences in creep propensities, but the use of such a short period herein is not to be construed as an endorsement of shortening the period for regular creep tests used for determination of design loads.

GRAIN-COARSENING EXPERIMENTS

In the previous report, steels with carbon contents of from 0.27 to 0.58 per cent carbon were heat treated to produce fine austenite grain size from 6 to 8 and coarse austenite grain size from 2 to 4 as measured by the A.S.T.M. method[6]. This spread in grain size was accompanied by significant differences in creep resistance of the various steels in the air-cooled condition.

In the work on the steels herein reported, grain sizes as fine as 6 to 8 were not always obtainable, so a fine austenite grain size of 5 to 7 and a coarse austenite grain size of 2 to 4 were produced in all steels as nearly as possible. These austenite grain sizes are the result of the combination of the times and temperatures used in the various heat treatments which are shown in Table II.

The best available discussion on the grain size of steel and its determination may be found in a recent paper prepared for the joint committee by Vilella of the U. S. Steel Corp.[7]

Most of the austenite grain sizes were determined using the gradient quench method in which small sections of the various steels were heated under the desired conditions of temperature and time and cooled by immersing in water about one-third to one-half of their length, allowing the remainder to cool in air. A longitudinal section was polished and etched and the austenite grain size determined either from the area which was martensitic with a network of fine pearlite outlining the austenite grains, or from the area which was pearlite with a network of ferrite outlining the austenite grains.

This method is very inconsistent with steels of the low carbon contents investigated but it was the best method available at the time most of the grain sizes were measured. Samples have since been obtained in which the austenite

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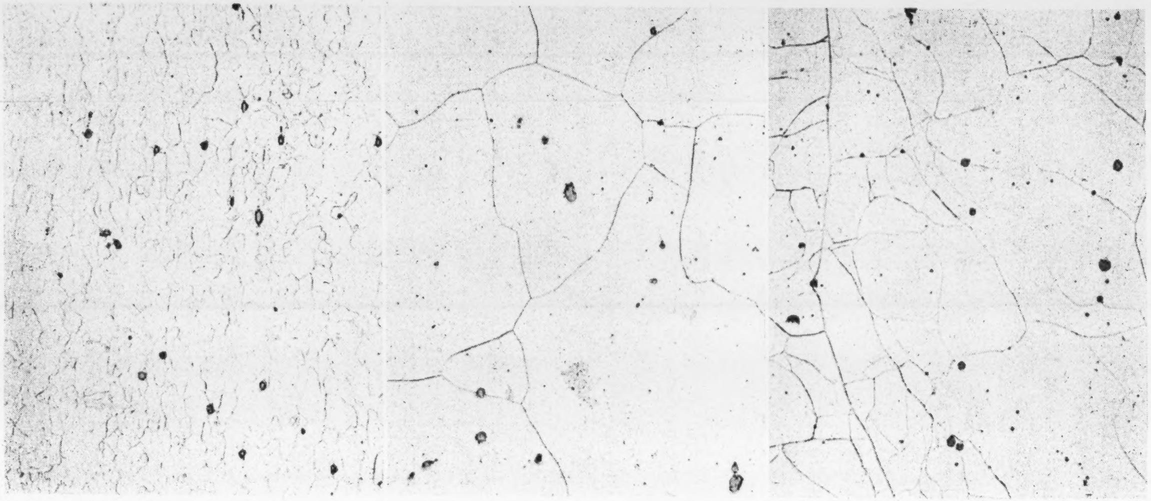
[3] H. W. Gillett and H. C. Cross, "Obtaining Reliable Values for Creep of Metals at High Temperatures," *Metals and Alloys*, Vol. 4, July, 1933, pp. 91 98.

[4] H. C. Cross, "High Temperature Tensile, Creep, and Fatigue Properties of Cast and Wrought High and Low Carbon 18 Cr-8 Ni Type Steel from Split Heats," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 56, No. 7, July, 1934, p. 533.

[5] 1939 Book of A.S.T.M. Standards, Part I, p. 1261.

[6] Tentative Classification of Austenite Grain Size in Steels (E 19 - 39 T), 1939 Book of A.S.T.M. Standards, Part I, p. 1175.

[7] J. R. Vilella, "The Grain Size of Steel," *Mechanical Engineering*, Vol. 62, No. 4, April, 1940, pp. 293-307.

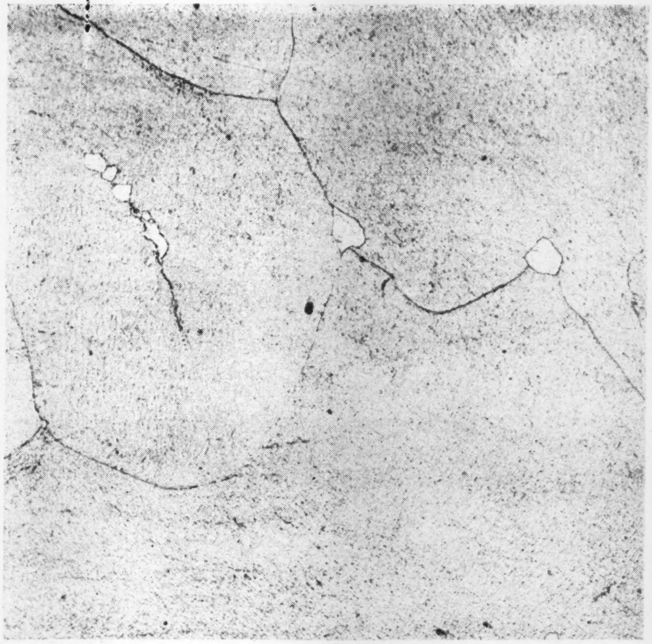


371-9-1 heated 15 min. in hydrogen at 1290 F., air cooled. Prior austenite grain size 5 to 8.

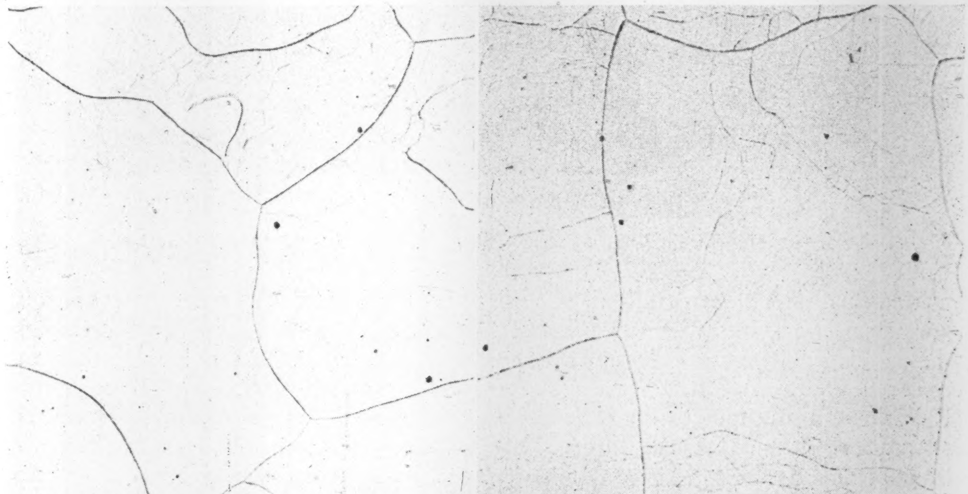
371-9-2A heated 10 hr. in hydrogen at 1830 F., air cooled. Prior austenite grain size 0 to 2.

371-9-2B heated 10 hr. in hydrogen at 1830 F., slow cooled. Prior austenite grain size 0 to 2.

FIG. 1.—Structure of Pure Iron as Tested, 0.003 per cent Carbon (× 100).



371-8-1 heated 15 min. in hydrogen at 1290 F., air cooled. Prior austenite grain size apparently very large.

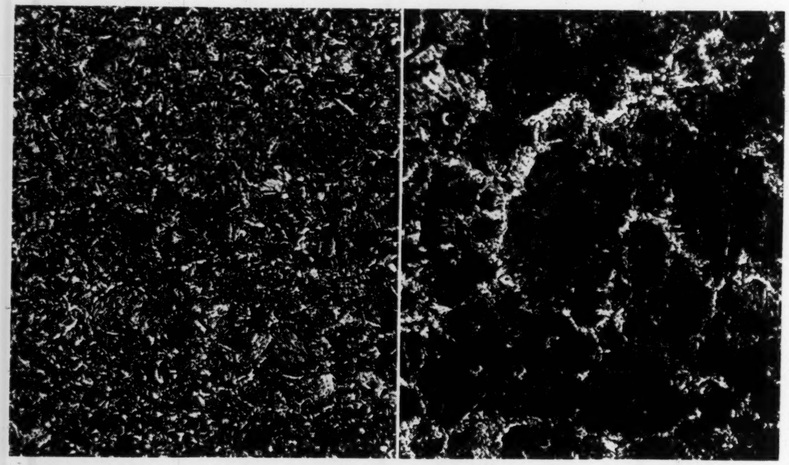


371-8-2A heated 10 hr. in hydrogen at 1830 F., air cooled. Prior austenite grain size 0 to 2.

371-8-2B heated 10 hr. in hydrogen at 1830 F., slow cooled. Prior austenite grain size 0 to 2.

FIG. 2.—Structure of Pure Iron Containing 0.5 per cent Silicon as Tested (× 100).

grains have been outlined by the heat etching method described by Day and Austin[8] and by Vilella[7] and in subsequent work this method will be used. Table II shows the heat treatments used to produce the desired austenite grain sizes of 5 to 7 and 2 to 4, the ranges chosen as fine and coarse for purposes of the tests herein reported. It is not considered necessary to detail the trial heat treatments used on the various steels before the desired grain sizes were obtained.



Heated 2 hr. at 1650 F. Prior austenite grain size 5 to 7.

Heated 2 hr. at 2000 F. Prior austenite grain size 2 to 4.

FIG. 3.—Prior Austenite Grain Sizes of Rimmed Steel, 0.06 per cent Carbon Core Material (× 100).

Metallographic examination of the pure iron 371-9 and the 0.5 per cent silicon iron 371-8 was made at the research laboratory of the Westinghouse Electric and Manufacturing Co. The structures obtained on the three 371-9 irons tested are shown in Fig. 1. Specimen 371-9-1 shows the fine ferrite structure produced by air-cooling of the recrystallized iron resulting from heating 15 min. at 1292 F. in hydrogen after cold forging from $1\frac{1}{8}$ in. to $\frac{7}{8}$-in. diameter (40 per cent reduction). Specimen 371-9-2A shows a very large ferrite grain size as heated at 1832 F. for 10 hr. in hydrogen. Notice that in this carbonless iron the Widmanstaetten structure usually resulting from rapid cooling of coarse austenite containing carbon was not obtained. However, there is no reason to suspect that the ferrite does not approximately indicate the prior austenite grain size. Specimen 371-9-2B shows the structure of the iron slow cooled after heating at 1832 F. for 10 hr. and the austenite grain size appears about the same as for the air-cooled specimen 371-9-2A. This specimen shows veining within the large ferrite grains such as is usually formed in slowly cooled unalloyed iron.

The structures obtained on the three 371-8 pure irons containing 0.5 per cent silicon are shown in Fig. 2. With regard to specimen 371-8-1 it seems strange that the severe cold work to which these specimens were subjected after the hot forging and high-temperature annealing should have failed to break down the large grains as examination of the cold-forged bars showed to be the case. The structures for 371-8-2A and 2B are very similar, both exhibiting veining to a marked degree.

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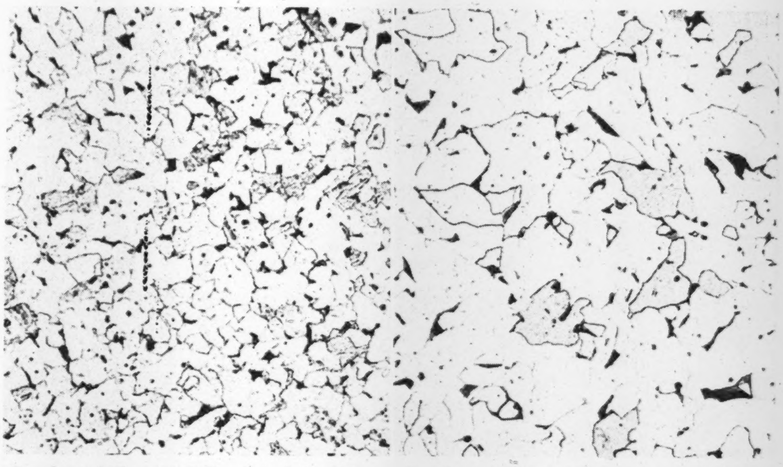
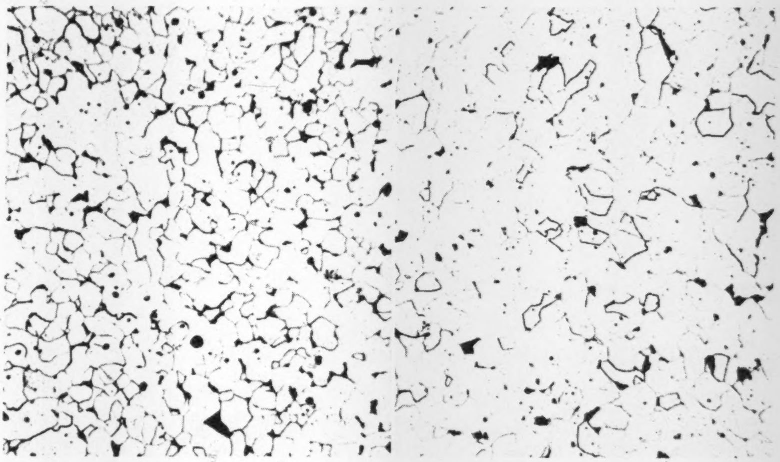
[8] M. J. Day and J. B. Austin, "Heat Etching as a Means of Revealing Austenite Grain Size," *Transactions*, Am. Soc. Metals (1939), Preprint 33.



Core heated 2 hr. at 1650 F., air cooled. Prior austenite grain size 5 to 7.

Core heated 2 hr. at 2000 F., air cooled. Prior austenite grain size 2 to 4.



Skin heated 2 hr. at 1650 F., air cooled. Prior austenite grain size 5 to 7.

Skin heated 2 hr. at 1825 F., air cooled. Prior austenite grain size 2 to 4, some 5.

Fig. 4.—Structures of Rimmed Steel, 0.06 per cent Carbon as Tested in Creep (× 100). Etched in 2 per cent Nital.

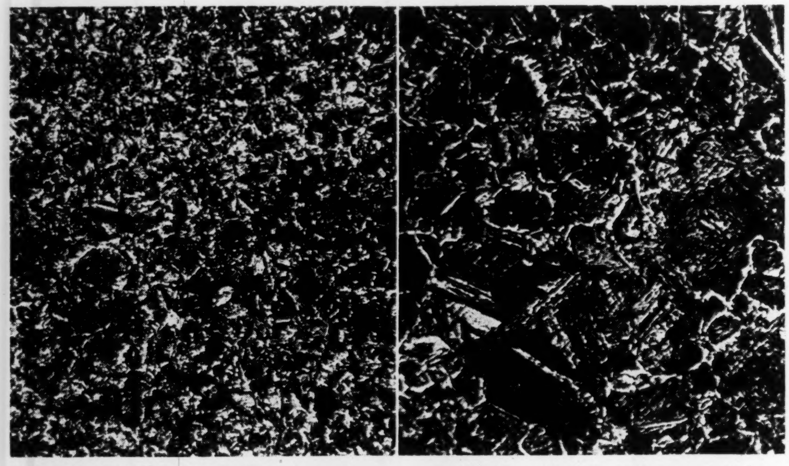
In the following discussion on steels the austenite and ferrite grain sizes resulting from the heat treatments will be shown in adjacent figures to facilitate description and to indicate the effect of austenite grain size and cooling rate on the actual structure as tested in creep.

Figure 3 shows the austenite grain sizes of the core of the rimmed steel with 0.06 per cent carbon as tested. Heating for 2 hr. at 1650 F. produced a grain size of 5 to 7 in both core and skin material, but a higher temperature was required for the core to coarsen to 2 to 4 than for the skin material. Since the austenite grain sizes of the skin material were similar, they are not shown, but are recorded in Table II.

Figure 4 shows the actual structures of the core and the skin of the rimmed steel as tested. The ferrite grain size of the coarsened core material is slightly larger than the ferrite of the material treated to produce fine-grained austenite. With the low carbon content of 0.06 per cent, the usual Widmanstaetten structure obtained on rapid cooling of material with coarse austenite grain is not so apparent. The ferrite grain size of the skin material is about the same whether obtained from either coarse or fine austenite.

Figure 5 shows the austenite grain size of the core of the silicon-killed steel with 0.08 per cent carbon as tested. Since the austenite grain sizes of the skin material were similar, they are not shown but are recorded in Table II. Heating at 1625 F. for 5 min. produced a grain size of 5 to 7 for both core and skin. Grain growth in the silicon-killed material is thus indicated to be much faster than in the rimmed material which required 2 hr. instead of 5 min. to produce a grain size of 5 to 7 at a temperature 25 F. higher. Likewise, lower temperatures were required to produce coarsening to 2 to 4 austenite grain size than for the rimmed steels in both the core and skin material.

Figure 6 shows the actual structures of the core and skin of the silicon-killed steel as tested. In both core and skin material, the ferrite grain size is slightly larger when obtained from coarse than from fine austenite.



Heated 5 min. at 1625 F. Prior austenite grain size 5 and 6, some 7.

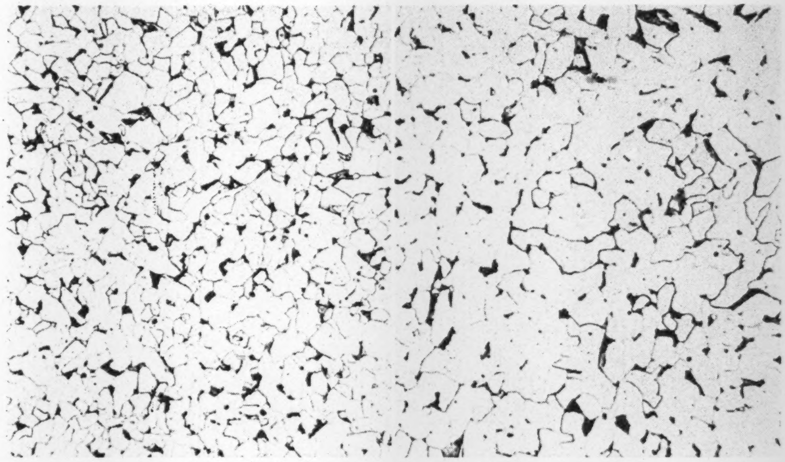
Heated 2 hr. at 1850 F. Prior austenite grain size 2 to 4, some 5

FIG. 5.—Prior Austenite Grain Sizes of Silicon-Killed Steel, 0.08 per cent Carbon Core Material (× 100).

The austenite grain sizes of the rimmed steel with 0.16 per cent carbon in the skin and 0.26 per cent carbon in the core are not shown, but the heat treatments used and grain sizes obtained are shown in Table II.

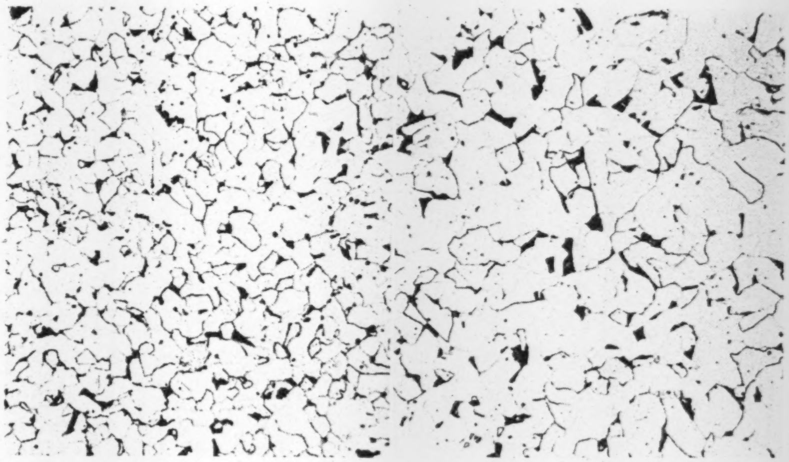
Figure 7 shows the actual structures of this higher-carbon rimmed steel as tested. The higher carbon content of the core material is apparent. With these higher carbon contents, the Widmanstaetten type of structure is obtained as air-cooled from the coarsening temperature.

Figure 8 shows the austenite grain sizes of the four S.A.E. 1015 steels as tested in the fine-grained condition. Steels 5247 (silicon-killed) and 3194 (silicon-aluminum-killed, 1 lb. of aluminum per ton) coarsen so readily that only 5 min. at 1575 F. produced the desired fine austenite grain of 5 to 7. Steel 5229 (2 lb. of aluminum per ton) was tested with a finer austenite grain size than the other steels, because of its tendency to form a few large grains when the temperature was raised above 1600 F., in an effort to produce 5 to 7 instead of the predominant grain size of 7 obtained in 10 min. at 1600 F. Steel 5280 (silicon-aluminum-killed, 2 lb. of aluminum per ton) was the steel with



Core heated 5 min. at 1625 F., air cooled. Prior austenite grain size 5 to 6, some 7.

Core heated 2 hr. at 1850 F., air cooled. Prior austenite grain size 2 to 4.

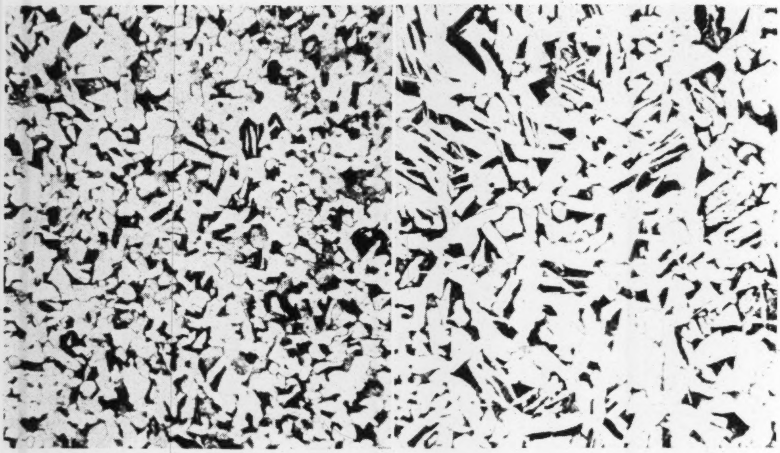


Skin heated 5 min. at 1625 F., air cooled. Prior austenite grain size 5 to 8, some 4.

Skin heated 3 hr. at 1750 F., air cooled. Prior austenite grain size 2 to 4, some 1 and 5.

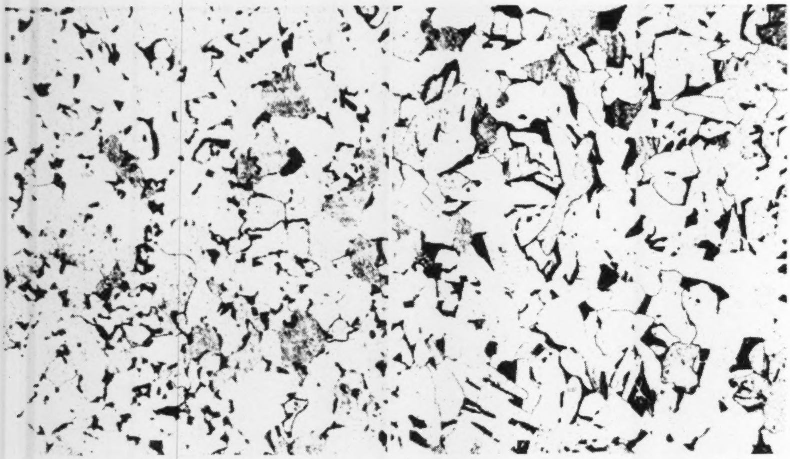
Fig. 6.—Structures of Silicon-Killed Steels, 0.08 per cent Carbon as Tested in Creep (× 100). Etched in 2 per cent Nital.

the greatest fine-grained tendencies, requiring 1 hr. at 1730 F. to produce an austenite grain size of 7 to 8.



Core heated 5 min. at 1560 F., air cooled. Prior austenite grain size 5 to 7.

Core heated 1 hr. at 2000 F., air cooled. Prior austenite grain size 2 to 4.



Skin heated 2 min. at 1560 F., air cooled. Prior austenite grain size 5 to 7.

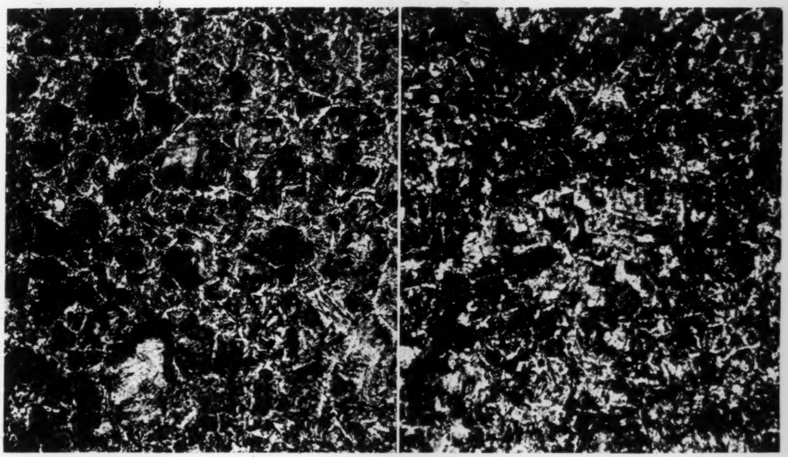
Skin heated 1 hr. at 1950 F., air cooled. Prior austenite grain size 2 to 4.

FIG. 7.—Structures of Rimmed steel as tested in Creep: Core, 0.26 per cent Carbon; Skin, 0.16 per cent Carbon (× 100). Etched in 2 per cent Nital.

All four steels were coarsened to approximately 2 to 4 grain size as shown in Fig. 9. Note that the silicon-killed steel (5247) required the lowest temperature for coarsening, the silicon-aluminum-killed steel (1 lb. of aluminum per ton—3194) the next highest, the aluminum-killed steel (5229) with 2 lb. per ton the next highest, and the silicon-aluminum-killed steel (2 lb. of aluminum per ton—5280) with 1 hr. at 2000 F. the highest.

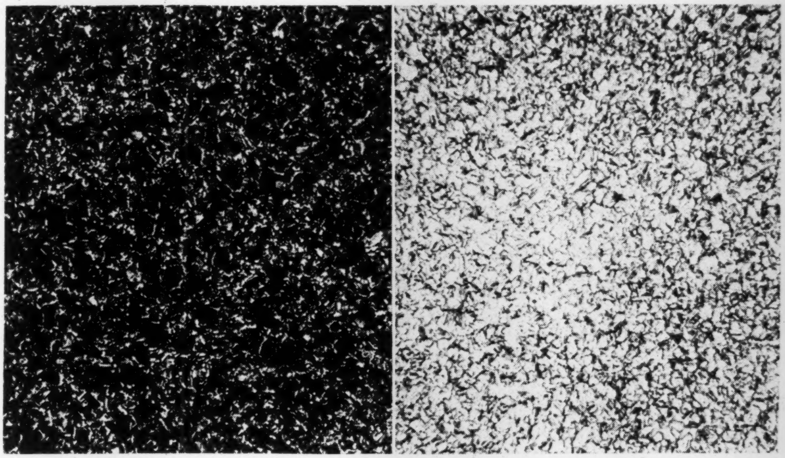
In addition to determining the way the austenite grain size correlates with the creep resistance of these four S.A.E.

1015 steels, the effect of rate of cooling from the coarsening temperature on the size and distribution of the ferrite and pearlite and the effect of the different structures obtained on the creep resistance has also been studied. All steels heat treated to produce fine-grained austenite were air cooled. Some specimens coarsened to 2 to 4 austenite grain size were air cooled from the coarsening temperature, while additional specimens were furnace cooled from the coarsening temperature to 1575 F., then furnace cooled at 40 F. per hour to 1250 F., then furnace cooled to room temperature. The resulting ferrite structures as subjected to creep test are shown in Figs. 10 and 11.



Steel 5247, silicon-killed, heated 5 min. at 1575 F. Prior austenite grain size 5 to 7, some 4.

Steel 3194, silicon-aluminum-killed, with 1 lb. of aluminum per ton added, heated 5 min. at 1575 F. Prior austenite grain size 5 to 7.

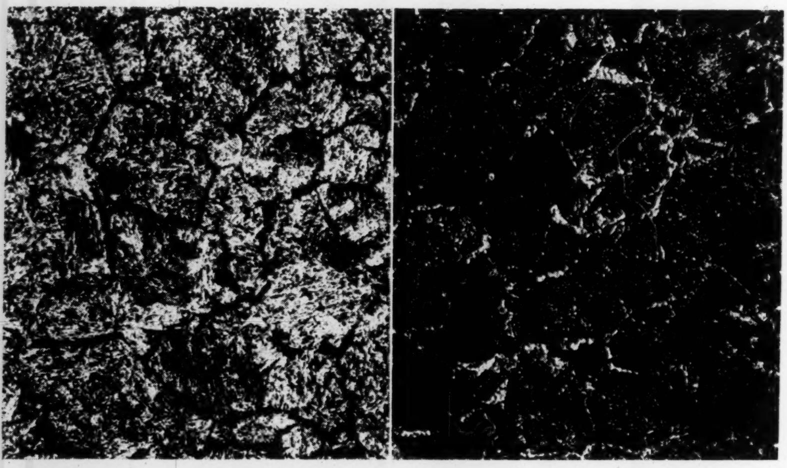


Steel 5229, aluminum-killed, with 2 lb. of aluminum per ton added, heated 10 min. at 1600 F. Prior austenite grain size 6 to 8.

Steel 5280, silicon-aluminum-killed, with 2 lb. of aluminum per ton added, heated 1 hr. at 1730 F. Prior austenite grain size 7 to 8.

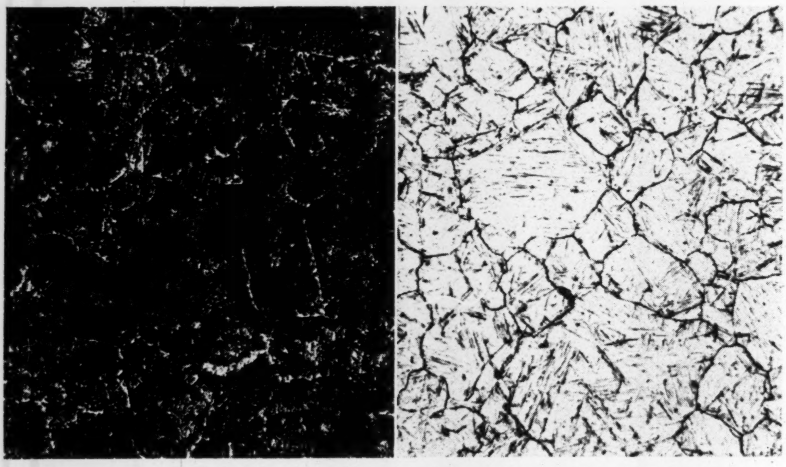
FIG. 8.—Prior Austenite Grain Sizes of S.A.E. 1015 Steels (Fine Grained) (× 100).

The marked similarity of the structures obtained from austenite of similar grain size is at once apparent. These specimens, treated to produce fine austenite and then air cooled, show fine pearlite distribution. Those specimens which were coarsened to a grain size of 2 to 4 and then air cooled show the pearlite areas in the familiar Widmanstaetten pattern which usually results from rapid cooling of coarse austenite grains. Those specimens which were coarsened and then slow cooled show very large pearlite areas evenly distributed in a large-grained and equiaxed ferrite matrix. The ferrite grain size of the coarsened and air-cooled specimens is smaller than that of the



Steel 5247, silicon-killed, heated 1½ hr. at 1800 F. Prior austenite grain size 2 to 4.

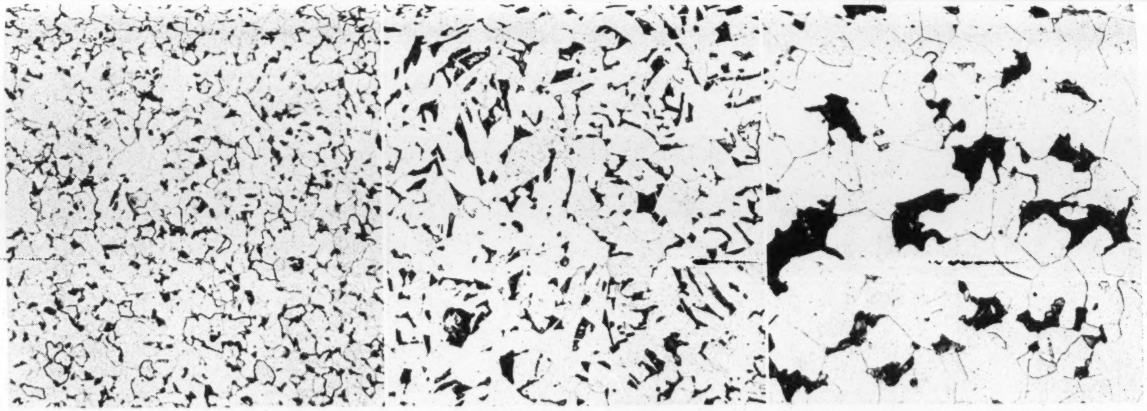
Steel 3194, silicon-aluminum-killed, with 1 lb. of aluminum per ton added, heated 2 hr. at 1900 F. Prior austenite grain size 2 to 4.



Steel 5229, aluminum-killed, 2 lb. of aluminum per ton added, heated 2 hr. at 1950 F. Prior austenite grain size 2 to 4.

Steel 5280, silicon-aluminum-killed, with 2 lb. of aluminum per ton added, heated 1 hr. at 2000 F. Prior austenite grain size 2 to 4, some 1 and 5.

FIG. 9.—Prior Austenite Grain Sizes of S.A.E. 1015 Steels (Coarse Grained) (× 100).

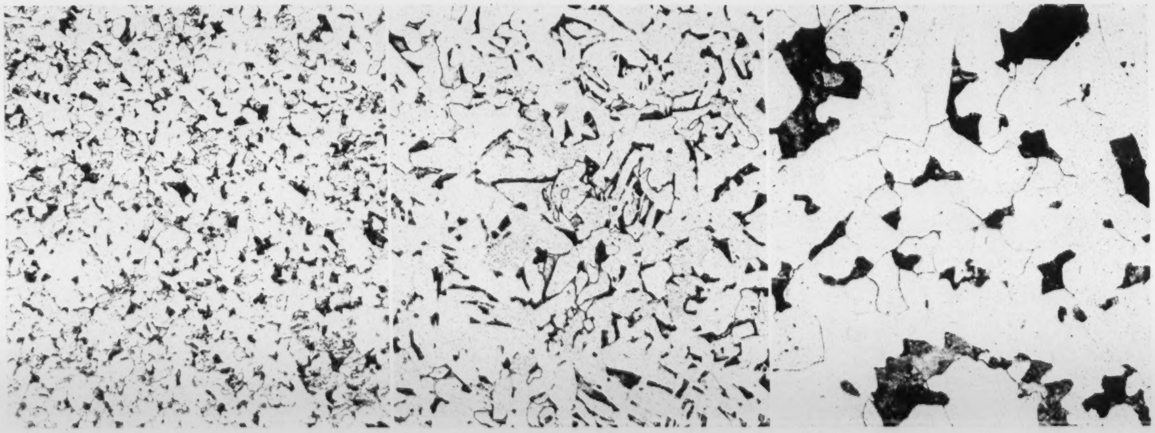


Steel 5247, silicon-killed.

Heated 5 min. at 1575 F., air cooled. Prior austenite grain size 5 to 7, some 4.

Heated 1½ hr. at 1800 F., air cooled. Prior austenite grain size 2 to 4.

Heated 1½ hr. at 1800 F., slow cooled. Prior austenite grain size 2 to 4.



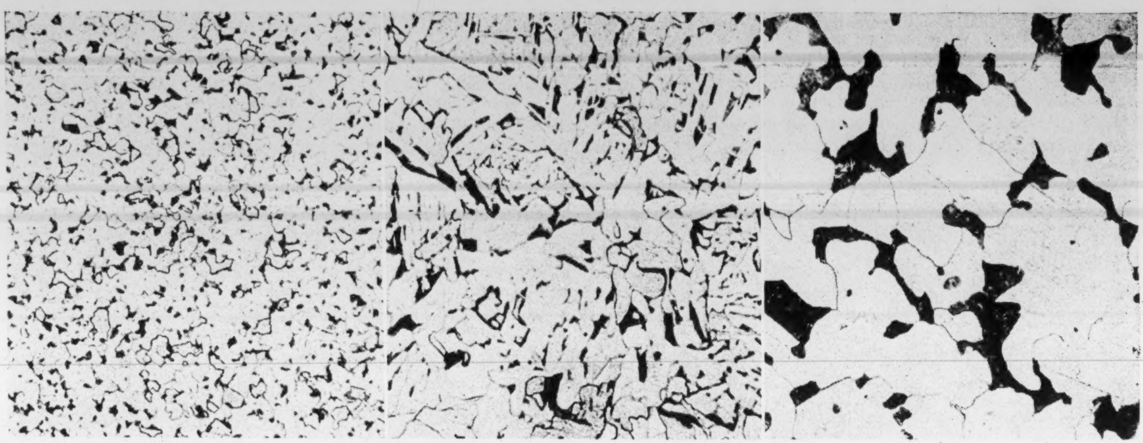
Steel 3194, silicon-aluminum-killed, 1 lb. of aluminum per ton added.

Heated 5 min. at 1575 F., air cooled. Prior austenite grain size 5 to 7.

Heated 2 hr. at 1900 F., air cooled. Prior austenite grain size 2 to 4.

Heated 2 hr. at 1900 F., slow cooled. Prior austenite grain size 2 to 4.

FIG. 10.—Structures of S.A.E. 1015 Steels 5247 and 3194 as Tested in Creep (× 100). Etched in 2 per cent Nital.

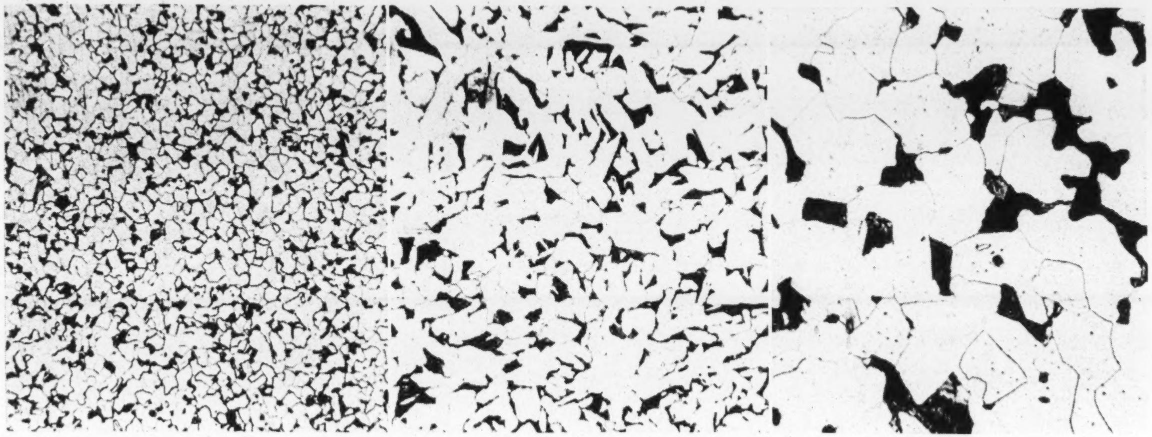


Steel 5229, aluminum-killed, 2 lb. of aluminum per ton added.

Heated 10 min. at 1600 F., air cooled. Prior austenite grain size 6 to 8.

Heated 2 hr. at 1950 F., air cooled. Prior austenite grain size 2 to 4.

Heated 2 hr. at 1950 F., slow cooled. Prior austenite grain size 2 to 4.



Steel 5280, silicon-aluminum-killed, 2 lb. of aluminum per ton added.

Heated 1 hr. at 1730 F., air cooled. Prior austenite grain size 7 to 8.

Heated 1 hr. at 2000 F., air cooled. Prior austenite grain size 2 to 4 some 1 and 5.

Heated 1 hr. at 2000 F., slow cooled Prior austenite grain size 2 to 4, some 1 and 5.

FIG. 11.—Structures of S.A.E. 1015 Steels 5229 and 5280 as Tested in Creep (× 100). Etched in 2 per cent Nital.

slow-cooled specimens but its relation with regard to the ferrite grain size of the air-cooled specimens with fine austenite is difficult to determine.

Room temperature tensile and impact properties have been determined for all of the steels as given the various heat treatments and these data will be discussed later in this report.

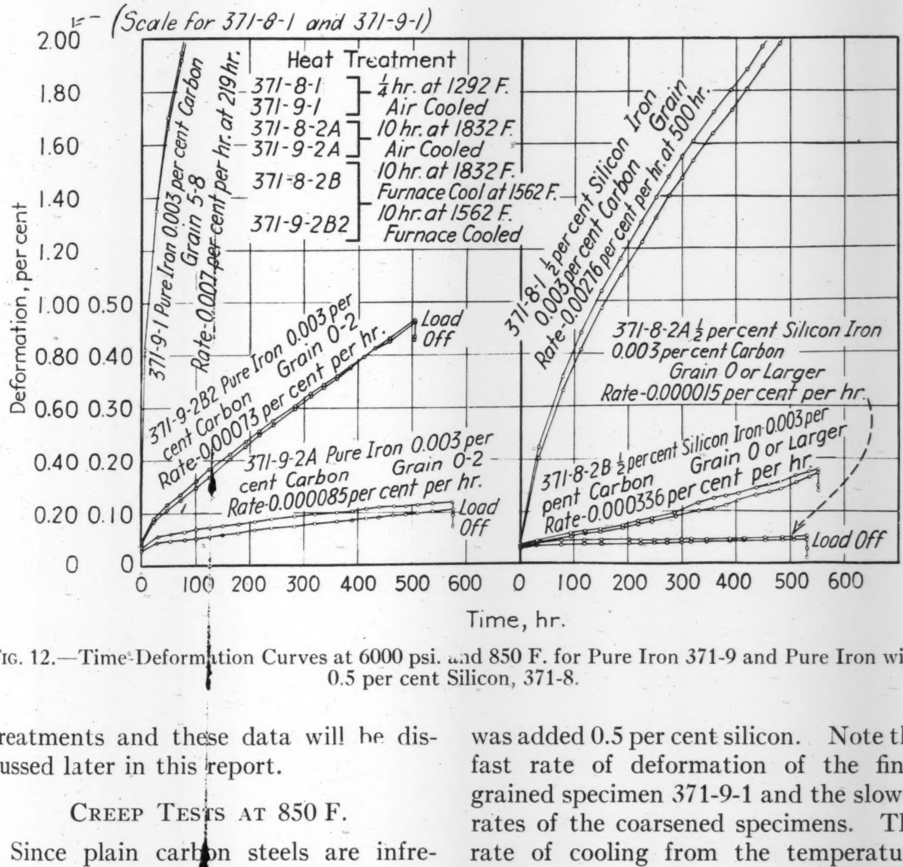


FIG. 12.—Time-Deformation Curves at 6000 psi. and 850 F. for Pure Iron 371-9 and Pure Iron with 0.5 per cent Silicon, 371-8.

CREEP TESTS AT 850 F.

Since plain carbon steels are infrequently used in high temperature service above 850 F., and since the previous tests[1] indicated similar effects of austenite grain size on creep resistance at 750 and 950 F. to those obtained at 850 F., all creep tests herein reported were made at 850 F. Their creep test data are shown in Table II, and a graphical summary of the effect of heat treatment and grain size on the creep rates of all the steels tested is shown in Fig. 19.

Figure 12 shows the beneficial effect accompanying coarse austenite grain size on the creep resistance of a practically carbon-free pure iron (0.003 per cent carbon) and a pure iron to which was added 0.5 per cent silicon. Note the fast rate of deformation of the fine-grained specimen 371-9-1 and the slower rates of the coarsened specimens. The rate of cooling from the temperature used to coarsen the iron also affects the creep resistance since the slow-cooled specimen (371-9-2B2) showed a rate almost nine times that of the air-cooled specimen (371-9-2A). The structures in Fig. 1 do not show any noticeable difference due to cooling rate, but the creep test shows a difference in properties. This effect of cooling rate in the absence of carbon is important, since

FIG. 11.—Structures of S.A.E. 1015 Steels 5229 and 5280 as Tested in Creep (× 100). Etched in 2 per cent Nital.

without these data similar effects of cooling rate on steels with 0.15 per cent carbon would likely be ascribed in part to the effect of the carbon present and its effect on the physical properties.

Figure 12 also shows three creep tests on the pure iron containing 0.5 per cent silicon (371-8) when heat treated similarly to the pure iron 371-9. The superior creep rate of the coarsened and air-cooled specimen 371-8-2A again demonstrates the beneficial effect accompanying coarse austenite grain size. Comparison of the creep rates of the two irons 371-9 and 371-8 (0.5 per cent silicon) shows that the addition of the 0.5 per cent silicon increased the creep resistance of the pure iron, both as fine and coarse grained. Even though the structure of specimen 371-8-1 air cooled after heating at 1292 F. was indicated to be coarse, the observed creep rate is as much faster than those specimens heated to higher temperatures (1832 F.) as would be expected if the material were fine grained. As with the pure iron without silicon, specimen 371-8-2B with 0.5 per cent silicon shows a faster rate of creep after slow cooling from 1832 F. than as air cooled from 1832 F.

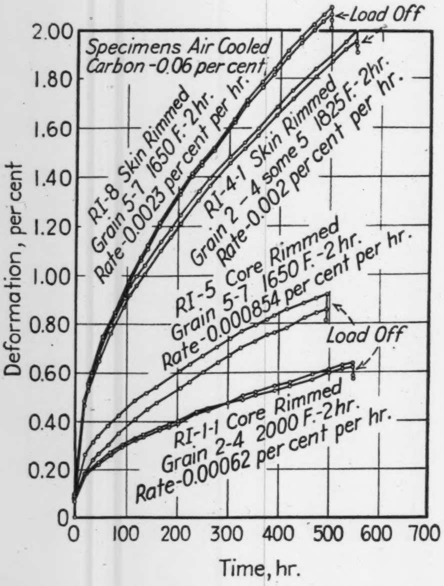


FIG. 13.—Time-Deformation Curves at 10,000 psi. and 850 F. for Rimmed Steel, 0.06 per cent Carbon.

These tests on the two pure irons indicate that the presence of carbon is not necessary for changes in austenite grain size to be accompanied by material changes in creep resistance. The possibility that the mode of existence of the carbon as affected by the various heat treatments is the sole cause of the differences in creep resistance is proved untenable.

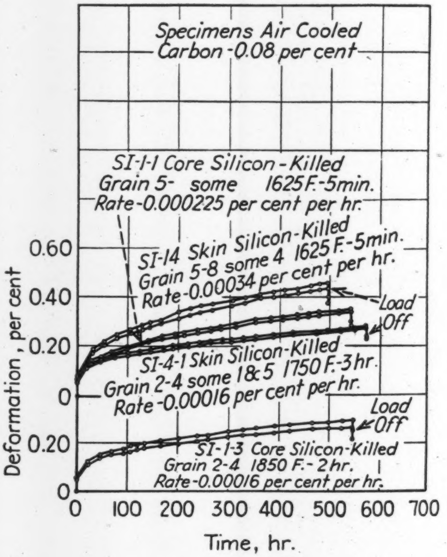


FIG. 14.—Time-Deformation Curves at 10,000 psi. and 850 F. for Silicon-Killed Steel, 0.08 per cent Carbon.

Figure 13 shows the time-deformation curves for the rimmed steel with 0.06 per cent carbon. The rimmed steel was tested at a higher load than the pure iron, so the creep rates are not directly comparable. As fine grained, the core material taken from the 4 by 4-in. rimmed steel billet shows better creep resistance than material taken from the skin of the billet. Coarsening of the austenite grain improves the creep resistance of both core and skin material slightly, but the improvement was less than is usually obtained by coarsening from a grain size from 5 to 7, to 2 to 4. The inferiority of rimmed steel in creep resistance as compared with silicon-killed steel is due to some other cause than austenite grain size, since the marked improvement usually accompanying the coarsening of steels of inferior creep resistance fails in the case of rimmed steels. Obviously the inferior properties are the result of the finishing practice peculiar to rimmed steel, but the exact cause is not apparent now. Several comments on this will be made later in the report after considering other data.

Figure 14 shows the time-deformation curves for the silicon-killed steel with 0.08 per cent carbon. Since deep etching of the 4 by 4-in. billet section showed a uniform structure, large differences in creep resistance between skin and core materials were not expected and were not obtained. Some improvement in creep resistance accompanied the coarsening of the austenite grain but the differences were not large. As coarsened to a grain size of 2 to 4 the core and skin materials (specimens SI-1-3 and SI-1-4) show the same creep rate.

Compared with the rimmed steel, the silicon-killed steel shows a superiority in creep rate as fine grained of 4 and 6 to 1 for core and skin materials and as coarse grained of 4 and 12 to 1.

Figure 15 shows the time-deformation curves for the rimmed steel with 0.16 per cent carbon in the skin and 0.26 per cent carbon in the core material. The effect of the difference of 0.10 per cent carbon content in improving the creep resistance is clearly shown by the lower creep rates of the core material as compared with the skin material. As in the case of the rimmed steel with 0.06 per cent carbon, coarsening produced only a small reduction of the creep rate. These different creep data for core and skin give an idea as to the variation in creep properties over a section that could be obtained in the use of high-carbon rimmed steel which is so susceptible to carbon segregation. The creep test data of this rimmed steel skin material with 0.16 per cent carbon are of value for comparing with data from the four killed steels of similar carbon content.

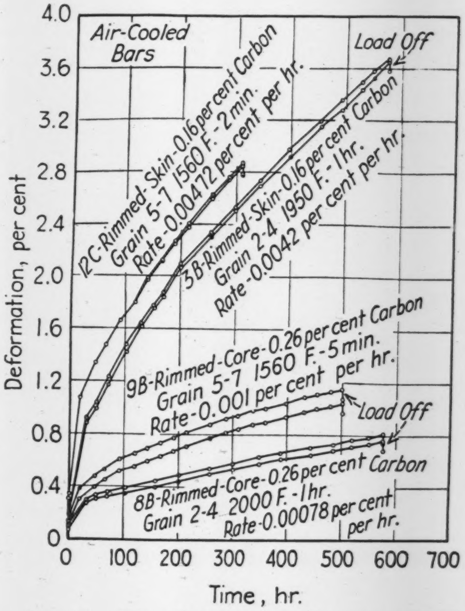


FIG. 15.—Time-Deformation Curves at 15,000 psi. and 850 F. for Rimmed Steel: Skin Material, 0.16 per cent Carbon; Core Material, 0.26 per cent Carbon.

Figure 16 shows the time-deformation curves of the four killed steels with 0.14 to 0.17 per cent carbon as air cooled from the temperatures which

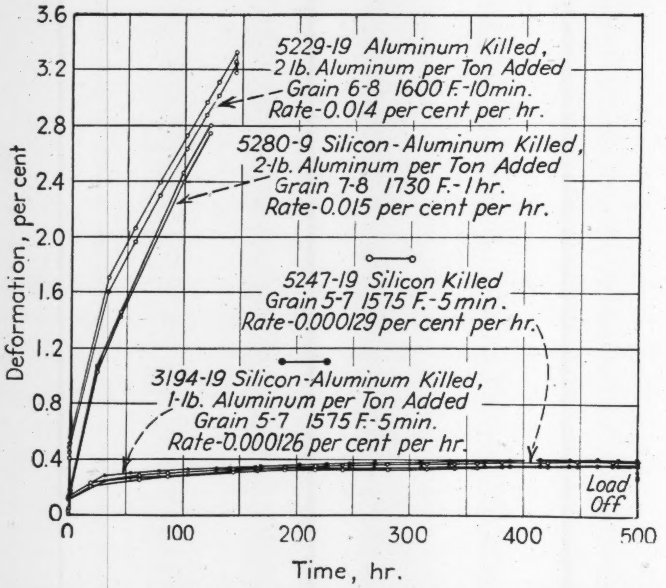


FIG. 16.—Time-Deformation Curves at 15,000 psi. and 850 F. for Killed S.A.E. 1015 Steels. Tested as air cooled from temperatures producing fine austenite grain size.

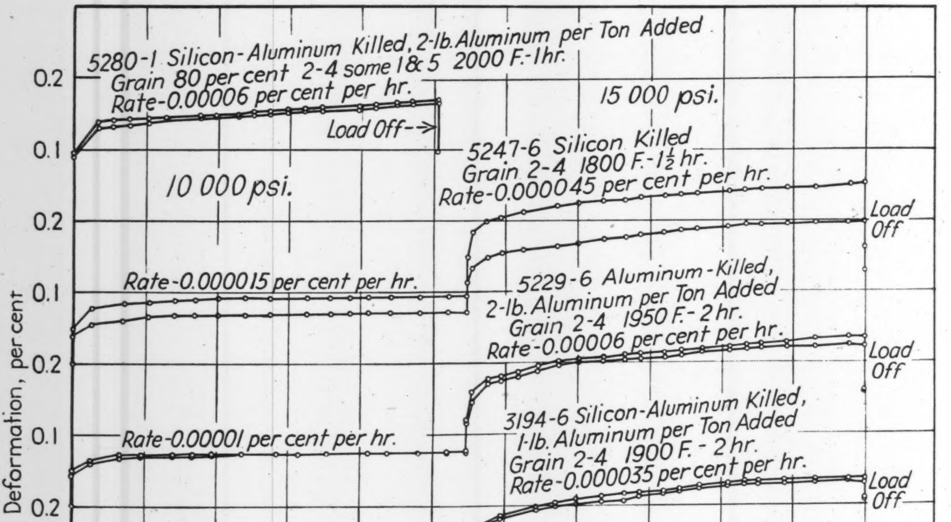


FIG. 17.—Time-Deformation Curves at 15,000 psi. and 850 F. for Killed S.A.E. 1015 Steels. Tested as air cooled from temperatures producing coarse austenite grain size.

produced a fine austenite grain size in each steel. At the load of 15,000 psi., the silicon-killed (5247) and silicon-aluminum-killed (1 lb. of aluminum per ton) steels (3194) shows similar low creep rates of about 0.00013 per cent per hour. The aluminum-killed steel (5229) and the silicon-aluminum-killed steel (5280) (2 lb. of aluminum per ton) with similar fine austenite grain size as the other two steels with no aluminum or low aluminun show a creep rate over 100 times greater. Apparently the addition of 2 lb. of aluminum per ton either in the absence of silicon or after the steel has been killed with 0.16 per cent silicon produces a steel of poor creep resistance in the fine-grained condition. Since steel with 1 lb. of aluminum per ton does not have poor creep resistance as fine grained, the effect begins with aluminum additions somewhere betwen 1 and 2 lb. per ton, in steel of the type represented by this series. The mechanism of this effect is not understood but it is probable that it is linked with the action of aluminum which gives a steel a fine-grained tendency and requires heating to higher temperatures to produce a substantial coarsening than is required in steels with lower aluminum additions.

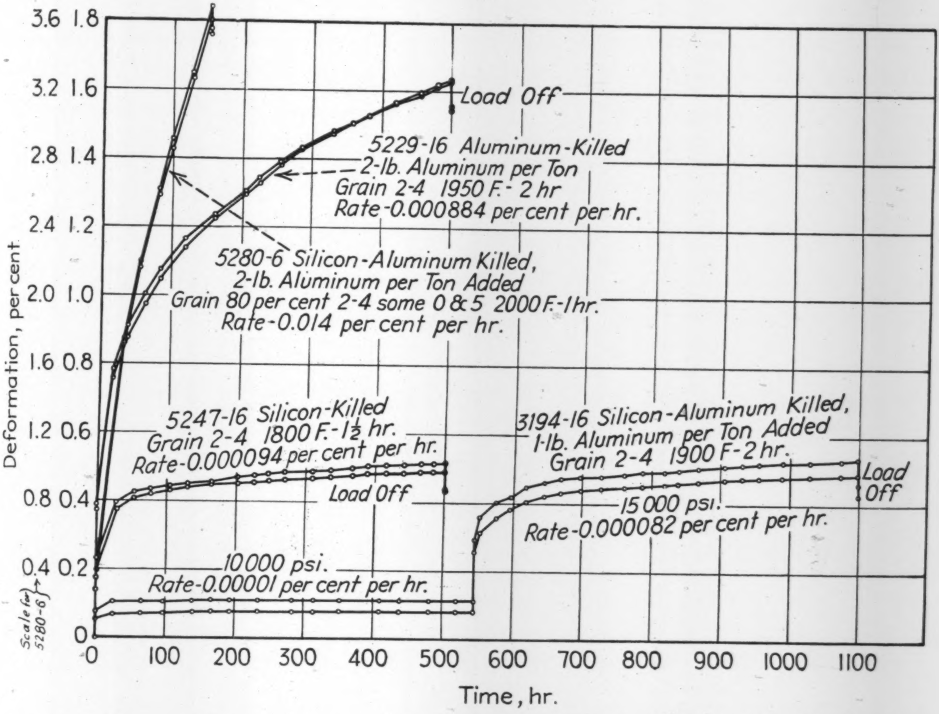


FIG. 18.—Time-Deformation Curves at 15,000 psi. and 850 F. for Killed S.A.E. 1015 Steels. Tested as slow cooled from temperatures producing coarse austenite grain size.

Figure 17 shows the time-deformation curves of the four killed steels as air cooled from temperatures which produced a coarse austenite grain size (2 to 4) in each steel. The first tests on steels 5247, 5229, and 3194 were run at 10,000 psi. for about 545 hr. The rates of deformation at this load were low so the loads were increased to 15,000 psi. and the tests continued for an additional 560 hr. Specimen 5280 was tested only at 15,000 psi. Coarsening of the austenite grain size followed by air cooling produced similar creep resistance in all four steels. The silicon-killed steel (5247) showed a rate of 0.000045 per cent per hour, the silicon-aluminum-killed steel with 1 lb. of aluminum per ton (3194) showed 0.000035 per cent per hour, while both the plain aluminum-killed steel (5229) and the silicon-aluminum-killed steel with 2 lb. of aluminum per ton (5280) showed a rate of 0.00006 per cent per hour. Even as coarsened, the steels with small or no aluminum additions show slightly better creep resistance than the other steels with 2 lb. per ton of aluminum. As compared with the creep rate as fine grained, steels 5247 and 3194 as coarsened show a reduction in rate of about 3 to 1. Steels 5229 and 5280 with 2 lb. of aluminum per ton as coarsened show reductions in creep rate of over 200 to 1. The heat treatment producing coarsening of the austenite grain size of the steels containing 2 lb. per ton of aluminum apparently eliminated the condition which contributes to their poor creep resistance in the fine-grained condition, while coarsening of the steels with none or 1 lb. of aluminum per ton only slightly improved the creep resistance which was already good in the fine-grained condition.

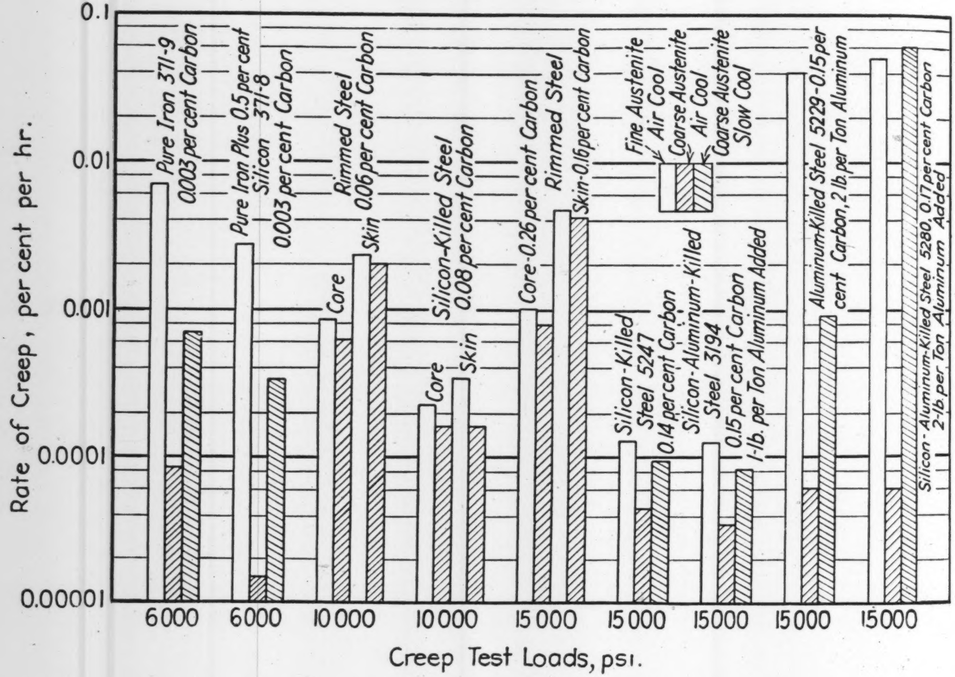


FIG. 19.—Effect of Heat Treatment and Resultant Austenite and Ferrite Grain on Creep Rates at 850 F.

Figure 18 shows the time-deformation curves of the four killed steels as slow-cooled from temperatures which produced a coarse austenite grain size (2 to 4) in each steel. Figures 11 and 12 show the Widmanstaetten structure obtained on air cooling of coarse austenite and the coarse ferrite and pearlite distribution obtained on slow cooling of coarse austenite. The slow-cooled silicon-killed steel (5247) and the silicon-aluminum-killed steel with 1 lb. of aluminum per ton (3194) show creep rates about double those shown as air cooled while the plain aluminum-killed steel shows a creep rate 15 times greater and the silicon-aluminum-killed steel with 2 lb. of aluminum per ton shows a creep rate over 250 times greater. Note that the greater increase in creep rate was obtained in the steel treated with 2 lb. of aluminum per ton which contained the silicon. This checks the usual observation that steels of increased fine-grained characteristics are produced by substantial additions of aluminum in those steels which have been previously deoxidized with silicon.

Attention is again called to Fig. 19 which permits a rapid appraisal of the relative effects of the heat treatments and grain size in the steels with different furnace practice and deoxidation on the creep rates at 850 F. The rate of creep is plotted on a logarithmic scale so that reference to the ordinate is necessary in comparing rates.

Discussion of Results

S.A.E. 1015 Steels:

The simultaneous effects of composition, deoxidation practice, heat treatment and its resultant austenite grain size and ferrite grain size produce creep test data that are difficult to explain when the test data for the whole series of irons and steels tested are considered.

Any hypotheses offered as possible explanations of the observed data must always consider the fact that coarsening of the austenite grain size of a very pure iron produced a significant increase in creep resistance and that this improvement was somewhat lessened by slow cooling rather than air cooling from the coarsening temperature. In addition to the condition imposed by the effect of changing only the grain size of the pure iron, any hypotheses must also explain the mechanism by which rimmed or aluminum-killed steels are poor in creep resistance as compared with silicon-killed steels in the fine-grained condition but possess also the property of inhibiting grain growth.

In the literature are conflicting statements and opinions regarding the role played by aluminum in inhibiting grain growth. Some investigators hold to the theory that aluminum forms nonmetallic inclusions that mechanically obstruct grain growth while others think the excess aluminum over that required for deoxidation, which is in solution in the ferrite, has either an effect on carbide solution in ferrite and austenite or a grain boundary effect, either or both of which inhibit grain growth.

Since larger differences in creep resistance were shown by the four S.A.E. 1015 steels with different deoxidation practices, these will be discussed first and any hypotheses developed from their consideration applied to the other steels later.

Two outstanding facts are to be noted. The two steels (5229 and 5280) treated with 2 lb. of aluminum per ton showed huge differences in creep resistance resulting from the various heat treatments used and previously described. The silicon-killed and the silicon-aluminum-killed steel, one without aluminum (5247) and the other with 1 lb. of aluminum per ton (3194) showed small differences in creep resistance due to the effect of the heat treatments and were consistently better than steels treated with 2 lb. of aluminum per ton.

It is apparent that the condition which accounts for the superiority of the silicon-killed steel or the silicon-aluminum-killed steel is little affected by drastic changes in heat treatment.

From the small effects on creep rates (3 or 5 to 1) of the addition of silicon to pure iron, it does not seem likely that the superior creep resistance of the silicon-killed and silicon-aluminum-killed steels (5247 and 3194) is due to the strengthening effect of silicon in ferrite. It seems much more possible that submicroscopic inclusions finely dispersed throughout the grains and formed from silicon (whatever their composition) are responsible for the superior creep resistance. Steels 5247 and 3194 showed a decrease in creep rate when coarsened, of about 3 to 1. Pure iron 371-9 showed a reduction in creep rate when coarsened of 80 to 1 and even though the pure iron was coarsened to a larger grain size, the effect of heat treatment was greater in this pure iron, thus illustrating even more strikingly the stability of the condition responsible for the good creep resistance of the silicon-killed steels. It would appear that even in this carbonless iron, some factors other than grain size are responsible for the large difference in creep resistance due to the heat treatment which produced coarsening of the austenite grain.

The poor creep resistance as fine grained of the steels treated with 2 lb. per ton of aluminum may be due to the size and location of the grain growth inhibitor present in the steel. Dorn and Harder[9] postulated that this grain-growth inhibitor in aluminum-killed steels was soluble in both ferrite and austenite, with the solubility increasing with temperature in either. That is, the idea is put forth that austenite and ferrite each have power to take into solid solution certain nonmetallics as the temperature rises, and to reject them from solution as the temperature falls. This is analogous to precipitation-hardening phenomena in general. Different nonmetallics would have different degrees of solubility and precipitability, and the solvent power of austenite and of ferrite would differ.

It will be recalled that the four steels furnished by the National Tube Co. were heated to 1650 F. for 1 hr. and air cooled. According to this hypothesis, the aluminum-treated steels when reheated and air cooled as in the heat treatments for fine austenite grain size as given in this report would reject grain growth inhibitor to the grain boundaries of the newly formed austenite, leaving some distributed within the grains. If the temperatures used to produce fine-grained austenite were not sufficiently high and the times insufficiently long to take a sufficient quantity of inhibitor into solution before air cooling, adequate separation of precipitate and consequent strengthening of the grains to slip would not occur, and inferior creep resistance, as compared with the silicon-killed steels, would result. Such a mechanism seems to fit the creep test data.

When these aluminum-killed steels are heated above their coarsening temperature to produce austenite grain size of 2 to 4, according to Dorn and Harder practically all grain growth inhibitor is taken into solution and when air cooled the inhibitor is precipitated dispersed throughout the grains. In this condition good creep resistance would be expected, and, as is shown in Table II and Fig. 9, such was the case. After the heat treatment which produced coarsening, all four of the steels showed approximately the same creep resistance.

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[9] J. E. Dorn and O. E. Harder, "Relation of Pre-Treatment of Steels to Austenitic Grain Growth," *Transactions*, Am. Soc. Metals, Vol. 26, p. 106 (1938).

As has been described, the four steels were also tested as slow cooled from the temperatures which produced coarsening. The silicon-killed steels with no or low aluminum show increases in creep rate as compared with the coarsened and air-cooled steels of about 2 to 1, a further indication of the stability of the condition contributing to their good creep resistance.

The plain aluminum-killed steel showed an increase in creep rate of about 15 to 1, but the rate was still much lower than was obtained in the fine-grained condition. The silicon-aluminum-killed steel treated with 2 lb. per ton of aluminum showed the same creep rate when slow cooled from the coarsening temperature as was obtained in the fine-grained condition. During this slow cooling from the coarsening temperature, it might be postulated that the grain growth inhibitor precipitates out in the grain boundaries and agglomerates, and since less inhibitor is dispersed within the grain poorer creep resistance is obtained.

It is natural to inquire why the addition of 1 lb. per ton of aluminum to steel 3194 after treating with silicon did not adversely affect its creep properties. Apparently this amount of aluminum was not sufficient to carry out the other functions of aluminum in combining with nitrogen, sulfur, and with oxygen in other forms, and leave enough to attack and eliminate submicroscopic silicon-containing inclusions which are postulated to contribute to the good creep resistance of the silicon-killed steels.

The addition of 2 lb. per ton of aluminum to steel 5280 completely eliminated the condition that gives silicon-killed steels its good creep resistance as fine-grained. Aluminum is a powerful deoxidizer and has a strong affinity for nitrogen and sulfur as well. It has been shown to attack and eliminate silicon-containing inclusions of a size that are visible. It is reasonable to assume then that aluminum will also eliminate the unseen silicon-containing inclusions which may account for good creep resistance.

At first it was thought that the poor creep resistance of the aluminum-killed steel 5229 might be due to the specific lack of silicon when the good properties of the other two silicon and silicon-aluminum-killed steels were considered. The study of steel 5280 which contained both silicon and aluminum shows that a sufficient quantity of aluminum wipes out the beneficial effects of the silicon when the steel is tested in the fine-grained condition.

No reasons can be offered for the better creep resistance as slow cooled from the coarsening temperature of the plain aluminum-killed steel (5229) as compared with the silicon-aluminum-killed steel (5280) also treated with 2 lb. of aluminum per ton, unless it is postulated that straight aluminum inclusions and more complex silicon-aluminum inclusions have different solubilities and are rejected in different fashion on cooling.

It should be clear from these creep test data obtained at comparable austenite grain sizes and with similar structures as tested that grain sizes as such are not the controlling factors in determining creep resistance but are the outward or pyrometric indication of the heat treatment given to the steel which produced the observed creep test effects. It will be recalled that, in work previously reported,[10] a fine-grained vanadium steel was shown to be highly creep resistant.

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[10] H. C. Cross and J. G Lowther, "Progress Report on Study of Effects of Manufacturing Variables on the Creep Resistance of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 149 (1938).

No doubt grain size as such is a contributing factor but there are indications that its effects may be minor rather than major. These relationships are emphasized when the austenite grain sizes shown in Figs. 8 and 9 and the actual structures as tested shown in Figs. 10 and 11 are re-examined. It should be obvious that any comparisons of creep resistance with austenite grain size should be made with a comparable cooling rate using similar size sections in all cases. The difference in structure between the air-cooled and slow-cooled coarsened S.A.E. 1015 steels are striking, yet the creep resistance for these two treatments in steels 5247 and 3194 differ very little and steels 5229 and 5280 differ widely. Certainly one would never suspect from examination of either the austenite or ferrite grain sizes of steels 5247 (silicon-killed) and 5280 (silicon-aluminum killed, 2 lb. of aluminum per ton) as shown in Figs. 8, 9, and 11 that such huge differences in creep resistance exist between them as are shown in Table II and Figs. 16 and 18. Neither would one suspect from the structures of steel 5280 as coarsened and slow cooled and as fine grained and air cooled (Fig. 11) that both show the same creep resistance.

Rimmed and Silicon-Killed Steels:

It has been shown that the silicon-killed steel with 0.08 per cent carbon possesses consistently better creep resistance than the rimmed steel with 0.06 per cent carbon and that coarsening and air cooling of the rimmed steel resulted in some improvement but not of the magnitude obtained in coarsening and air cooling of steels treated with 2 lb. of aluminum per ton. As in the silicon-killed S.A.E. 1015 steels, the lower carbon silicon-killed steel possesses good creep resistance little improved by heat treatment producing coarsening, again indicating stability of the cause of the better properties as compared with the rimmed steel. The higher temperatures required for coarsening of the rimmed steel suggest that in the absence of appreciable quantities of aluminum some other material is reacting to inhibit grain growth, which however does not produce as fine a dispersion of grain inhibiting material when rapidly cooled from the coarsening temperature and hence not as good resistance to deformation within the grains. The silicon-killed steel shows slightly higher tensile and yield strengths and slightly lower ductility and impact resistance as compared with the rimmed steel.

The higher carbon rimmed steel with 0.16 per cent carbon in the skin and 0.26 per cent carbon in the core likewise show little improvement by rapid cooling from the coarsening temperature. It is of interest to compare the creep resistance of the skin of this rimmed steel (0.16 per cent carbon) with the S.A.E. 1015 steels heat treated to the same austenite grain sizes both fine and coarse. In the fine-grained condition this rimmed steel is inferior to the silicon-killed steel 5247 (no aluminum) and the silicon-aluminum-killed steel 3194 (1 lb. of aluminum per ton) but superior to the two steels 5229 and 5280 treated with 2 lb. of aluminum per ton. As air cooled from the temperature producing coarsening this rimmed steel is much inferior to any of the four killed steels.

It should be pointed out that Jenkins, *et al.*[11] reported an acid open-hearth steel "E" of 0.19 per cent carbon, 0.40 per cent manganese, 0.08 per cent silicon of poor creep resistance as normalized, which was not improved by the coarsening process applied by these authors. A piece of this steel, supplied through the courtesy of C. H. Desch, was analyzed for aluminum by the U. S. Steel Corporation Research Laboratory. The analyses show no indication of any aluminum addition. Deep etching shows no core such as would appear in a rimmed steel. Thus this steel appears to be a killed or semi-killed steel, of suitable silicon content for good creep resistance rather than the poor creep resistance reported. This case is mentioned to show that something else than presence of silicon and absence of aluminum in too great amounts, and something else than grain size, are involved in creep behavior.

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[11] C. H. M. Jenkins, H. J. Tapsell, G. A Mellor, and A. E. Johnson, "Some Aspects of the Behavior of Carbon and Molybdenum Steels at High Temperatures," *Transactions*, Chem. Eng. Conference of the World Power Conference (London), Vol. 1, pp. 122-162 (1936).

TABLE III.—ROOM TEMPERATURE PHYSICAL PROPERTIES OF THE FINE-GRAINED AND COARSE-GRAINED STEELS.

Steel Designation and Specimen Number		Carbon, per cent	Heat Treatment	Austenite Grain Size A.S.T.M. Number	Yield Point, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Charpy Impact, ft-lb.
RI	Rimmed steel—core....	0.06	1650 F., 2 hr.	5 to 7	39 275	49 700	45.8	74.4	56
RI	Rimmed steel—core....	0.06	2000 F., 2 hr. air cool	2 to 4	32 875	48 675	45.5	73.8	54
RI	Rimmed steel—skin....	0.06	1650 F., 2 hr.	5 to 7	37 775	45 925	49.5	80.2	57
RI	Rimmed steel—skin....	0.06	1825 F., 2 hr.	2 to 4 some 5	34 225	44 440	49.3	79.9	59
SI	Silicon-killed—core....	0.08	1625 F., 5 min.	5 to 6 some 7	39 800	52 800	45.0	73.4	55
SI	Silicon-killed—core....	0.08	1850 F., 2 hr. air cool	2 to 4	35 940	51 950	44.8	71.4	53
SI	Silicon-killed—skin....	0.08	1625 F., 5 min.	5 to 8 some 4	42 300	52 100	45.0	72.5	55
SI	Silicon-killed—skin....	0.08	1750 F., 3 hr.	2 to 4 some 1 & 5	31 675	51 375	44.2	71.0	54
9B	Rimmed steel—core....	0.26	1560 F., 5 min.	5 to 7	44 400	62 425	41.0	65.3	38
8B	Rimmed steel—core....	0.26	2000 F., 1 hr. air cool	2 to 4	36 660	61 800	40.7	65.0	34
12C	Rimmed steel—skin....	0.16	1560 F., 2 min.	5 to 7	31 500	50 000	49.0	74.8	56
3B	Rimmed steel—skin....	0.16	1950 F., 1 hr.	2 to 4	32 300	50 050	48.0	74.2	56
5247	Silicon-killed..........	0.14	1575 F., 5 min., air cool	5 to 7 some 4	41 000	58 415	42.0	69.3	51
5247	Silicon-killed..........	0.14	1800 F., 1½ hr., air cool	2 to 4	34 125	58 250	41.3	68.7	51
5247	Silicon-killed..........	0.14	1800 F., 1½ hr., then[a]	2 to 4	34 190	55 575	30.8	62.6	25
3194	Silicon-aluminum killed 1 lb. of aluminum per ton.........	0.15	1575 F., 5 min., air cool	5 to 7	41 675	59 175	42.3	70.5	53
3194	Silicon-aluminum killed 1 lb. of aluminum per ton.........	0.15	1900 F., 2 hr., air cool	2 to 4	36 790	58 700	41.0	68.8	51
3194	Silicon-aluminum killed 1 lb. of aluminum per ton.........	0.15	1900 F., 2 hr., then[a]	2 to 4	28 750	52 975	33.5	62.8	22
5229	Aluminum-killed—2 lb. per ton..............	0.15	1600 F., 10 min., air cool	6 to 8 mostly 7	37 975	54 500	45.3	72.4	53
5229	Aluminum-killed—2 lb. per ton..............	0.15	1950 F., 2 hr., air cool	2 to 4	29 775	55 500	42.5	68.7	45
5229	Aluminum-killed—2 lb. per ton..............	0.15	1950 F., 2 hr., then[a]	2 to 4	22 725	50 000	39.5	63.3	23
5280	Silicon-aluminum killed—2 lb. of aluminum per ton.........	0.17	1730 F., 1 hr., air cool	7 to 8	47 400	60 525	42.7	70.6	51
5280	Silicon aluminum killed—2 lb. of aluminum per ton.........	0.17	2000 F., 1 hr., air cool	80% 2 to 4 some 1 & 5	36 800	60 625	41.0	66.4	41
5280	Silicon-aluminum killed—2 lb. of aluminum per ton.........	0.17	2000 F., 1 hr., then[a]	80% 2 to 4 some 1 & 5	27 175	54 850	39.5	61.2	32

[a] Furnace cooled from maximum temperature to 1575 F., then cooled 40 F. per hour to 1250 F., then furnace cooled.

On the hypothesis that some silicon-containing nonmetallic constituent is needed, the presence of the oxygen, nitrogen, sulfur, or whatever other element or elements enter the nonmetallic inclusion, is as important as the silicon itself. The presence and availability of such other elements will depend upon the steel-making technique used. Unfortunately, the British workers do not know the melting history of their steel, so that finding out what not to do in order to keep from producing steel like the British specimen remains a matter for experiment.

ROOM TEMPERATURE PHYSICAL PROPERTIES OF THE STEELS AS FINE GRAINED AND COARSE GRAINED

Table III shows the available room temperature physical properties of the various steels tested. Due to the limited quantity of material available, no room temperature physical properties were obtained on the carbonless irons as tested in creep.

Coarsening of the austenite grain size which produced only small changes in creep resistance for the rimmed steel and silicon-killed steel with 0.06 and 0.08 per cent carbon likewise produced only minor changes in physical properties. Some lowering of the yield strengths resulted, but the tensile strengths, ductility, and impact resistance were little affected by coarsening and were quite similar for both steels.

For the rimmed steel with core of 0.26 per cent carbon and skin of 0.16 per cent carbon coarsening produced little change in creep or physical properties except yield strength for the core material, but in the skin material better creep as coarsened was accompanied by no change in physical properties.

As heat treated to produce both fine- and coarse-grained skin material, this rimmed steel with 0.16 per cent carbon shows slightly lower strength, higher ductility and impact resistance than the four S.A.E. 1015 steels while its creep resistance as fine grained was better than steels 5229 and 5280 treated with 2 lb. of aluminum per ton.

As heat treated to produce the fine-grained condition, all four S.A.E. 1015 steels show about the same tensile strength, elongation, reduction of area, and impact resistance while steel 5280 which was silicon-aluminum killed (2 lb. of aluminum per ton) shows a yield strength higher than the other three. Yet this steel 5280 with the best tensile and yield strength was the poorest in creep resistance. As air cooled from the coarsening temperature, the tensile strengths for all four steels are about the same and similar to the values as fine grained. The yield strengths are all lower, the ductilities are about the same, and the impact resistance of the steels with 2 lb. of aluminum per ton (5229 and 5280) is lower, yet the four steels are all better in creep resistance than in the fine-grained condition. As slow cooled from the coarsening temperature, the tensile strengths are similar and slightly lower than for the air-cooled fine or coarse-grained condition; the steels with 2 lb. of aluminum per ton added show somewhat lower yield strengths and impact resistance but higher elongations than the other two steels. These data show that the creep resistance of the various steels given the various heat treatments could not be predicted from their room-temperature physical properties.

It should be noted that although in several cases coarsening followed by slow cooling produced lower impact resistance as compared with the other treatments, the lowest value obtained was 22 ft-lb.—still quite high enough not to be considered lacking in shock resistance.

SUMMARY

Creep tests at 850 F. on a very pure almost carbon-free iron have shown that air cooling from a temperature producing appreciable coarsening of the austenite grain results in marked improvement in creep resistance and some but lesser improvement as slow cooled.

Similar heat treatments of a pure iron containing 0.5 per cent silicon produced similar improvements in creep resistance and the iron containing the silicon showed the better creep resistance.

Heat treatments producing coarsening of a rimmed steel (0.06 per cent carbon) and a silicon-killed steel (0.08 per cent carbon) resulted in small improvements in creep resistance. The silicon-killed steel was consistently superior to the rimmed steel.

A higher carbon rimmed steel showed marked carbon segregation with 0.16 per cent carbon in the skin and 0.26 per cent in the core. Coarsening was of little effect in the core material, but considerable improvement in creep resistance resulted from coarsening of the skin material.

Four S.A.E. 1015 steels with different final additions were tested as air cooled from a temperature producing fine austenite grain and as both air cooled and slow cooled from a temperature producing coarse austenite grain.

In the fine-grained condition, the silicon-killed steel and the silicon-aluminum-killed steel with 1 lb. of aluminum per ton added are far superior in creep resistance to the two steels containing 2 lb. of aluminum per ton whether with or without silicon.

As air cooled from the coarsening temperature, all four S.A.E. 1015 steels show similar creep resistance. As slow cooled from the coarsening temperature the two steels containing 2 lb. of aluminum per ton are again inferior to the two silicon and silicon-aluminum-killed steels with none or 1 lb. of aluminum per ton. It is apparent that the effect of aluminum in amounts of 2 lb. per ton is to eliminate or markedly reduce the good creep resistance of silicon-killed steels.

These data clearly indicate that austenite grain size is not the only variable affecting creep resistance and that it may be only the external indication of the maximum temperature of heat treatment which has caused other changes not indicated by the structures to occur. Any complete discussion of the effect of heat treatment and austenite grain size on creep resistance requires inclusion of the mode of cooling from the heat treatment temperatures used.

Widely differing creep resistance was obtained in several of the steels, and these differences could not be predicted from a study either of their structures or their physical properties as tested in tension.

The creep test continues to be one of the most sensitive means of detecting differences between steels apparently of similar properties when tested in other ways. This report concerns progress in a long-time program of the joint committee in which study of the relative effects of variables on creep resistance will be continued. Among these variables, grain size, carbon content and distribution, and the postulated submicroscopic nonmetallic constituents, appear to deserve special consideration.

DISCUSSION

Mr. A. Nadai.[1]—I wish to commend the authors for this most important report on the influence of metallurgical factors on the creep of steel. By observing the creep rates in tension tests, powerful means become available for the studies of steels. It seems that *the creep rates* offer the new mechanical quantities which may serve for determining quantitatively the effects of heat treatments and some of the obscure and not yet better understood phenomena which occur later in metals under stress at elevated temperatures.

Another point that I should like to mention refers to whether the authors believe that creep in some of their investigated materials does not so much occur in the grain boundaries but in the interior of the grains. Silicon was mentioned as one of the important materials present influencing the creep of steels. This points to the fact that it would be interesting to investigate further the influence of some of the minutely distributed constituents, whether present in the boundaries or in the interior of the grains. I think that such work as the authors have done throws important light on questions in which engineers and metallurgists are interested, namely, what are the observable changes in the structure of a metal, what is the mechanism of these changes while a metal creeps, or more probably what are the *various* mechanisms associated with the slow continuous flow of the metals under stress at elevated temperatures; several things happen at the same time in the complex structure of an alloy which need to be distinguished as the possible causes contributing to continuous flow.

The creep problem is an example of a complex group of questions. Although I am interested more in the mechanical end of it and in the laws which must connect stress with rate of strain, with strain and with time, I think that it should be realized that various types of flow exist according to the processes occurring in the structure, about which much information similar to that furnished by the authors is still needed.

Mr. J. M. Lessells.[2]—It is interesting to note that the authors have been continuing investigations on the same material as used by Mr. Jenkins in England. All those who are interested in rationalizing creep data were rather disconcerted with the findings of Jenkins since the variations found seemed to indicate the hopelessness of such a task. The authors now appear to be able to explain the inconsistencies found by Mr. Jenkins and I should like to ask whether he has seen the present paper since his observations on the authors' interpretation would be of interest.

Mr. Bradley Stoughton.[3]—If I differ with the authors in one or two points, I trust I may be forgiven. They speak of the fact that the use of 0.5 per cent silicon in almost carbonless iron would not be responsible for the increase in the creep rate because of the silicon strengthening the ferrite. That metal is almost pure ferrite, and we do not know how much silicon will strengthen ferrite at 850 F., but it seems to me that a great deal of that strengthening is due to the silicon strengthening the ferrite; furthermore the authors will notice, that when you have the high silicon the iron does not begin to stretch even at the very beginning, indicating that it is not strain hardening that has precipitated something in there and lessened creep, but that the material itself does not begin to stretch the way it does in every other case. There is one other point which I should like to bring up: I do not know whether the authors intended it or not, but it seems to me that it would be a mistake at this stage to try to minimize the effect of grain size by saying that the effect of grain size on creep resistance may be merely an indication of something else. I believe that it is almost universally accepted that a large grain size, whether obtained by high heating or because the steel does not have inherent small grain size, gives a better creep resistance than a steel with a small grain size. At a meeting some time ago the question of grain size was brought up and it was stated that where the temperature was below the equicohesive temperature, the grain size might not be important but where it was above the equicohesive temperature the grain size was a large factor.

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[3] Professor of Metallurgical Engineering, Lehigh University, Bethlehem, Pa.

I know that the authors did not say definitely that grain size was not important in itself, but I would dislike to see that factor minimized. It seems to me it is of very great importance in connection with creep resistance.

MR. FRANCIS B. FOLEY.[4]—I wonder how the authors support their precipitation theory on the basis of the results obtained with pure iron. They have not said what does precipitate, but here we have this pure iron with rapid cooling and coarse grain, producing the best creep rate, air cooling producing an intermediate creep rate, and slow cooling producing a rapid creep rate. There is nothing to support the idea that the rate of cooling is bringing about a precipitation. I suppose that the idea is that in rapid cooling the precipitant is kept in solution and not precipitated at the temperature of creep testing.

MR. H. W. GILLETT.[5]—There was one piece of evidence presented 2 yr. ago that I think should be recalled. We brought that evidence in very intentionally, early in the game, so that we would not go off half-cocked on this idea that grain size was everything. That evidence is the fact that a fine-grained vanadium steel is good in creep.

We are basing the present working hypothesis, which is only a hypothesis, partly on the fact that in vanadium steel the fine-grained metal has good creep resistance and is only a little improved by coarsening.

We are all accustomed to recognize vanadium as a hardening element. We know about secondary hardness in high-speed steel and plain vanadium steel, and we also know, from the use of vanadium for strengthening, that it has the habit of coming out where you want it, probably within the grain rather than at the grain boundary.

Another strong point in this argument is the very nice work Mr. Miller and coworkers at the U. S. Steel Corp. laboratories did on molybdenum steel; they showed actual signs of a "peppering" or precipitation through the grain in a creep-resistant steel—not the most creep resistant, but quite creep resistant—and they noted that if they used a heat treatment that would be expected to give some submicroscopic, invisible, inclusions, that was still better than when they agglomerated them into the form visible as "pepper," so the whole thing seems to tie up and be logically explained on the basis of just plain precipitation hardening, except for the one English steel. Now let us think a little more about it. Most of our creep-resistant steels seem to be strengthened by some kind of a carbide. The carbides tend to agglomerate and spheroidize. What is spheroidization but one of the precipitation-hardening effects? If, in the nonmetallic material present in steel we have a material which is only slightly soluble, less soluble than carbide, and agglomerable only with difficulty, perhaps if we put the right kind of nonmetallic dirt in our steel, we will have a creep-resistant steel that you cannot spheroidize and cannot weaken. If the hypothesis has any correctness at all, and if we find out how to put in a nonmetallic material which will go into and out of solution in the proper way, perhaps we can produce a more stable steel for general creep resistance than we have at the present time. Possibly we want to consider the intentional production of the right type of soluble and precipitable nonmetallic material. This hypothesis is certainly incomplete—it may be incorrect—but it should serve as a working guide to use in finding out whether things fall in line or not.

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[4] Superintendent of Research, The Midvale Co., Philadelphia, Pa.

[5] Metallurgist, Battelle Memorial Inst., Columbus, Ohio.

MR. R. W. EMERSON.[6]—After hearing this paper, I have come to the conclusion that in order to produce better creep properties in steel, air cooling is to be preferred to furnace cooling, and a coarse grain size (2 to 4) is preferable to one which is fine (6 to 8). This raises two questions in my mind.

The present Tentative Specifications for Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 40 T)[7] specify or perhaps infer that the pipe shall be furnace cooled from 1700 F. at a rate not to exceed 50 F. per hr. I wonder whether this is correct, or whether we should be normalizing (air cooling) the pipe from the coarsening temperature.

The authors have shown that a grain size of 2 to 4 imparts better creep properties to steel than when the steel is of exceedingly fine grain (6 to 8). Specifications A 206 call for a grain size of 3 to 6 after annealing from 1700 F. Seldom, if ever, does a No. 3 grain develop with only a minimum number of No. 4 being obtained after a 1700 F. coarsening treatment.

Approximately 75 per cent of the material will be made up of Nos. 5 and 6, the remaining material consisting of grain size Nos. 4 and 7. If it would be advantageous to obtain a more coarse-grained material than is generally obtainable under the existing Specifications A 206 – 40 T, I believe it will be necessary to use a coarsening temperature somewhat above 1700 F.

MR. P. G. MCVETTY.[8]—In reporting this investigation, the authors have set a standard which other investigators should attempt to follow, in trying to give, as clearly as possible, all of the factors which might influence the interpretation of the results. Correlation of creep data from different sources is difficult under the most favorable conditions, and this paper indicates reasons for some of the discrepancies which have been observed. The tests of pure iron specimens of similar structure in which slow cooling gave a creep rate almost nine times that obtained after air cooling indicate the importance of knowing the complete past history of test specimens. As this investigation has progressed, it has brought out the need for extreme care in the use of creep data for design purposes. It is not sufficient to specify analysis, heat treatment, and structure, but deoxidation practice and other factors must be closely controlled.

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[6] Metallurgist, Pittsburgh Piping and Equipment Co., Pittsburgh, Pa.

[7] 1940 Supplement to Book of A.S.T.M. Standards, p. 252.

[8] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

Design for high-temperature service must be based on reliable test data and accumulated experience. The choice of materials and working stresses depends fundamentally upon the assumption that the material put into service is essentially similar to that for which information is available. Further study of the effects of variables is the most promising basis for improvements in specifications.

MR. H. C. CROSS[9] (*Author's closure*).—With regard to Mr. Nadai's comments, we have felt that when the steel is heated to a temperature sufficiently high to produce coarsening, whatever grain-inhibiting material was present had been taken into solution; otherwise coarsening would not have occurred. It is postulated that rapid cooling (probably air cooling is sufficient) produces a precipitation of the material which inhibits grain growth, dispersed throughout the grains and thus contributes to good creep resistance but slower cooling gives time for inclusions to agglomerate and migrate to the grain boundaries, in which case they would be less effective in producing creep resistance.

As suggested by Mr. Lessells, a copy of this report has been forwarded to Mr. Jenkins in England for his comments.

With regard to Mr. Stoughton's comments, the addition of 0.5 per cent silicon improves the creep resistance of the pure iron, but the improvement is only four or five to one in creep rate. Therefore we do not feel that the addition of silicon and its strengthening effect on ferrite is sufficient to account for the differences in creep rate of 150 to 200 to 1 between the silicon-killed steel and the aluminum-killed steel, particularly since the two silicon-aluminum-killed steels which differed only in the aluminum additions also showed widely differing creep rates in the fine-grained condition.

It was not intended to minimize the effect of grain size but rather to give a strong warning that grain size as such is not the sole or principal controlling factor in determining creep resistance. It is so easy to remember a simple broad statement that coarse grain size gives better creep resistance than fine grain size. It was to prevent the use of such a statement that our comments were made in the paper. Steels of the same coarse grain do not always give the same creep resistance and fine- and coarse-grained structures of the same steel may show the same creep resistance. For carbon steels it has been shown that composition, deoxidation practice, grain size, and heat treatment are all controlling factors and any statement concerning creep resistance which takes into account only grain size effects may be entirely incorrect and misleading.

In answer to Mr. Foley, the pure iron supplied by Westinghouse was electrolytic iron melted under dry hydrogen and no analysis other than 0.03 per cent carbon was supplied. It is assumed that it was very pure iron, carefully prepared. There is no explanation for the effects of the various heat treatments on the creep rates for this pure iron, but the effects are similar to those found for the steels. Further work on this pure iron and other pure irons is needed to explain these effects of heat treatment.

We do not have data which will permit a statement as to whether the present tentative specifications for heat treatment of Seamless Carbon-Molybdenum

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[9] Metallurgist, Battelle Memorial Inst., Columbus, Ohio.

Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 40 T)[7] produce the best obtainable creep resistance. Our work to date has been only on plain carbon steels. Alloy steels may not react similarly. There is some work being done elsewhere on carbon-molybdenum steel which may serve to answer the question raised by Mr. Emerson. A grain size of 3 to 6 would be a mixed grain size. A few experiments on the effect of mixed grain size indicated the creep resistance was improved in about the same proportion as the amount of coarse grains present. Better creep resistance might be obtained in carbon-molybdenum steels if a heat treatment was used that produced a more uniformly coarsened steel than 3 to 6 or even coarser grain size, but other objectionable properties might result. Only experiments would develop such information.

Mr. McVetty's comments follow closely our own conception of the project and the possible gains to be derived. We need to know all of the variables that affect the creep resistance of steels. Knowledge of the variables involved will permit choice of dependable design loads and the development and use of better creep-resistant materials.

APPENDIX II

LIST OF PUBLISHED REPORTS WITH REFERENCE TO TESTS ON 0.35 PER CENT CARBON STEEL K20

(PROJECT No. 22)

PREPARED BY P. E. MCKINNEY, A. B. BAGSAR, E. L. ROBINSON, AND J. S. WORTH

This list of published reports containing references to tests on 0.35 per cent carbon steel (K20) includes 33 items numbered approximately in chronological order. They may be classified according to the following subjects:

Method of manufacture and physical properties	1, 32
Long-time constant-stress creep tests	2, 6, 10, 15, 19, 27, 29
Low-temperature impact tests	4, 33
Cooperative check creep tests	9, 20, 25, 26, 27, 28, 29
Constant strain rate tests	11
Relaxation tests	12, 13, 18, 23
Strength discrepancies and manufacturing variables	16, 22
Short-time rupture or acceptability test	21, 24
Variable velocity impact tests	3, 5, 7, 17, 30
Electrical resistivity test	31
Editorial reference	26, 28
Fatigue tests	1, 34

(1) Short-Time Tensile Tests at 850 F. of the 0.35 per cent Carbon Steel Material K20, Progress Report by Subgroup D on Short-Time Tensile Tests, H. J. Kerr, Chairman, Joint Research Committee on Effect of Temperature on the Properties of Metals, *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, February, 1936, RP-58-4, p. 97.

Gives details of manufacture, chemical composition and physical properties.

(2) H. C. Cross and F. B. Dahle, "Long-Time Creep Tests of 18 Cr 8 Ni Steel and 0.35 per cent Carbon Steel," *Ibid.*, RP-58-3, p. 91.

Gives constant stress test results on K20 at 850 F. as follows:

10000 psi. for 1000 hr.
9000 psi. for 1400 hr.
8000 psi. for 4900 hr.
7500 psi. for 4000 hr.

(3) H. C. Mann, "The Relation Between the Tension Static and Dynamic Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 323 (1935).

Gives effect of notch length on unit absorption of energy in impact specimens and effect of velocity on energy absorption.

(4) R. Sergeson and S. W. Poole, "General Summary and Comparison of Procedure and Results Obtained on Cooperative Study of Low-Temperature Impact Testing of 0.35 per cent Carbon Steel (K20)," Appendix I, Progress Report of Subcommittee P, Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 132 (1936).

(5) H. C. Mann, "High Velocity Tension Impact Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 85 (1936).

Gives tension impact properties under various velocities of loading and effect of heat treatment upon critical impact properties.

(6) H. C. Cross and F. B. Dahle, "Report on Long-Time Creep Tests of 18 per cent Chromium 8 per cent Nickle Steel and 0.35 per cent Carbon Steel," Appendix II, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 143 (1936).

Gives constant stress test result at 850 F. for 9600 hr. at 8000 psi. and for 8700 hr. at 7500 psi.

(7) G. F. Jenks, "Application of Tension Impact Test," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, p. 313; Vol. 60, p. 95.

Gives impact tests with long and short notches at room and low temperatures, comparisons of tension impact tests and notched bar tests at low temperatures, and effect of tempering treatments upon critical impact velocities.

(8) A. Nadai, "Creep of Metals," *Proceedings*, London Congress, Internat. Assn. Testing Mats., April 24, 1937. Group A—Metals, Sub-Group I, p. 4. This is a reference to (18) and (19).

(9) C. L. Clark, "Cooperative Creep Tests on 0.35 C. Steel K20 at 850 F. and 7500 psi.," Progress Report of Subcommittee E, Committee III, Joint Research Committee on Effect of Temperature on the Properties of Metals, *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, July, 1937, RP-59-4, p. 439.

Gives preliminary results of constant stress tests in 8 laboratories at 7500 psi. and 850 F.

(10) Howard C. Cross and J. G. Lowther, "Long-Time Creep Tests of 18 Cr. 8 Ni. Steel K19 and 0.35 C Steel K20," *Ibid.*, RP-59-5, p. 441.

Gives constant stress test results on K20 at 850 F. as follows:

8000 psi. for 9600 hr., then unloaded and reloaded. 7500 psi. for 12,200 hr.

(11) A. Nadai and E. A. Davis, "Constant Strain-Rate Tests on 0.35 C. Steel K20 at 850 F.," Contributed by Subcommittee E, Committee III, Joint Research Committee on the Effect of Temperature on the Properties of Metals, *Ibid.*, RP-59-6, p. 447.

Five constant speed tests run at rates from 0.166 per cent per hour up to 3900 per cent per hour. (Note that these are high speeds 1,000,000 to 100,000,000 times design rates.)

(12) Ernest L. Robinson, "A Relaxation Test on 0.35 C Steel K20," *Ibid.*, RP-59-7, p. 451.

Gives test results for 3700 hr. at 850 F. under following conditions:

Constant extension of 1.5 mils per in.
Constant stress of 7500 psi.
Constant stress of 5500 psi.
Constant stress of 4000 psi.

(13) N. L. Mochel, "Relaxation Tests on 0.35 C. Steel K20 at 850 F.," *Ibid.*, RP-59-8, p. 453.

Gives residual stresses after periods of time from 50 to 1000 hr. and with initial stresses of 5000, 10,000 or 20,000 psi.

(14) J. B. Romer, "Further Reports Annual Meeting December 1936,"—Appendix of Preprint; A.S.M.E. Transactions, July 1937.

Editorial comment.

(15) Howard C. Cross and J. G. Lowther, "Report on Long Time Creep Tests of 18 per cent Chromium, 8 per cent Nickel Steel and 0.35 per cent Carbon Steel," Appendix III, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I, p. 178 (1937).

Continues constant stress test results at 850 F. and 7500 psi. to 15,400 hr.

(16) H. W. Gillett, "Discrepancies in the Load-Carrying Abilities of Carbon Steels at 850 F.," Appendix IV, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Ibid.*, p. 187.

Creep strength of other carbon steels runs as high as two to three times that of K20.

(17) H. C. Mann, "A Fundamental Study of the Design of Impact Test Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 102 (1937).

Gives relation of energy absorption and notch volume in impact specimens, and effect of temperature on energy absorption and transition velocity.

(18) A. Nadai and J. Boyd, "Relaxation of Steels at Elevated Temperatures—A New Automatic Relaxation Machine," *Journal of Applied Mechanics*, Vol. 5, September, 1938, p. A-118.

Abstract only of paper which contains results of five relaxation tests at 850 F. of 15 to 300-hr. duration and initial stresses of 3990 to 18,500 psi. Also gives results of reloading tests.

(19) Howard C. Cross and J. G. Lowther, "Report on Long-Time Creep Test of 0.35 per cent Carbon Steel (K20)," Appendix II, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 121 (1938).

Gives final results of 20,400-hr. constant stress test at 850 F. and 7500 psi. Also gives rates for 1000 hr. each at 800, 840, 860 and 900 F., to determine temperature effect.

(20) C. L. Clark, "Report Covering Creep Tests on 0.35 per cent Carbon Steel (K20) at 850 F. Under a Stress of 7500 psi.," Project No. 14, Appendix III, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Ibid.*, p. 130.

Gives final results up to 3000 hrs. of constant stress tests in eight laboratories at 7500 psi. and 850 F. Includes several tests at somewhat different temperatures.

(21) A. E. White and C. L. Clark, "Report on Acceptability Tests for High Temperature Characteristics," Project No. 17, Appendix IV, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Ibid.*, p. 142 (1938).

Gives 15-hr. rupture tests at 650, 750, 850 and 1000 F.

(22) H. C. Cross and J. G. Lowther, "Progress Report on Study of Effects of Manufacturing Variables on the Creep Resistance of Steels," Project No. 18, Appendix V, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Ibid.*, p. 149.

Shows that creep strength of K20 at 850 F. could be more than doubled by high temperature normalizing treatment and consequent coarsening of austenitic grain size.

(23) Ernest L. Robinson, "The Resistance to Relaxation of Materials at High Temperature," Progress Report of Project 16, Joint Research Committee on the Effect of Temperature on the Properties of Metals, *Transactions*, Am. Soc. Mechanical Engrs., Vol. 61, August, 1939, p. 543.

Gives relaxation results obtained by three different methods of test in as many laboratories and comparison with constant stress results.

(24) C. L. Clark and A. E. White, "Report on Further Experiments with a Proposed Acceptance Test; Effect of Grain Size," Project No. 17, Appendix II, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 225 (1939).

(25) B. B. Betty, "Creep Test at 850 F. and 7500 psi.," International Nickel Co., Inc., Huntington, W. Va.

3000-hr. test. Copy of detailed laboratory report on file.

(26) G. R. Brophy, "Two Creep Tests at 850 F. and 7500 psi.," International Nickel Co., Inc., Bayonne, N. J.

Both tests still in progress.

(27) J. B. Romer, "Creep Test at M. I. T. at 850 F. and 7500 psi.," Babcock & Wilcox Co.

This test has run approximately 33,000 hr. and is still in progress.

(28) P. G. McVetty, "Two Creep Tests at 850 F.," Westinghouse Electric and Manufacturing Co. These were not included in (20), but may be had upon request.

(29) "Creep Test at 7500 psi., 850 F.," originally reported in (20) by one of the cooperating laboratories but still continuing. Has run more than 26,000 hr.

(30) J. F. Rodenhauser, "Effect of Speed of Impact on Path of Rupture in Steel," Thesis submitted to Massachusetts Institute of Technology.

Col. G. F. Jenks has one copy which may be borrowed for examination.

(31) M. E. Warnant, "Determination of Electrical Resistivity of K20 Steel," University of Brussels, Brussels, Belgium. Report promised upon completion.

(32) Room Temperature Tensile Test Before and After Creep Tests, (reported in (20)) with photomicrographs and grain size ratings, also statement of having sent bar stock to J. J. Kanter.—Letter from R. H. Aborn, U. S. Steel Corp. on file.

(33) Battelle Memorial Institute report to Republic Steel Corp. dated February 29, 1936, covering Effect of Temperature upon the Impact Properties of K20 Steel. Copy on file.

This was part of cooperative work of Subcommittee P and is included in condensed and edited form in (4).

(34) H. W. Russell and W. A. Welcker, Jr., "Damage and Overstress in the Fatigue of Ferrous Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 118 (1936).

Showed K20 material to have comparatively high notch sensitivity and no resistance to damage by overstress.

REPORT OF COMMITTEE B-1

ON

COPPER AND COPPER-ALLOY WIRES FOR ELECTRICAL CONDUCTORS

Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors held two meetings during the year: one in Atlantic City, N. J., on June 29, 1939, and one in New York, N. Y., on April 9, 1940.

Subsequent to the 1939 annual meeting, Committee B-1 presented to the Society, through Committee E-10 on Standards, Tentative Specifications for Soft Rectangular and Square Copper Wire for Electrical Conductors (B 48 - 39 T) to replace immediately the Standard Specifications for Soft Rectangular Copper Wire (B 48 - 38). This recommendation was accepted[1] by Committee E-10 on August 24, 1939, and the new tentative specifications appear in the 1939 Book of A.S.T.M. Standards.[2]

Several additions and changes in the membership of the committee have been made during the year. Committee B-1 now consists of 17 producer, 15 consumer, and 2 general interest members, making a total of 34, represented on the committee by 40 individuals. Twelve additional individuals, not members of Committee B-1, augment this membership in subcommittee work.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II (P. H. Grogan, chairman) and *Subcommittee VII* (R. S. Pratt, chairman) are continuing their studies on the effect of speed of testing in collaboration with Committee E-1 on Methods of Testing.

Subcommittee III (T. S. Fuller, chairman) completed its work on the packaging section of the Specifications for Soft or Annealed Copper Wire (B 3 - 39) and has been discharged.

Subcommittee IV (J. R. Becker, chairman) is recommending a revision of the Tentative Specifications for Soft Rectangular and Square Copper Wire for Electrical Conductors (B 48 - 39 T), as given in detail later in this report. This subcommittee will continue to study desirable changes in these specifications.

Subcommittee V (P. H. Grogan, chairman) is working on a tentative revision of the Standard Specifications for Hot-Rolled Copper Rods for Electrical Purposes (B 49 - 39) to provide for a twist test.

Subcommittee VIII (W. H. Bassett, Jr., chairman) is preparing Specifications for Bare Rope-Lay-Stranded and Bunch-Stranded Soft Copper Cables for Electrical Conductors, working in cooperation with a group of the Insulated Power Cable Engineers Association. The Standard Specifications for Bare, Stranded Copper Cable (B 8 - 39) cover the concentric-lay constructions, and it was thought at one time that they might be expanded to include other constructions. It is the present thought to restrict the scope of Specifications B 8 to the concentric-lay-stranded construction and to promulgate new specifications covering rope-lay-stranded cables composed of concentric-lay strands and also of bunch-stranded strands. In addition, it seems desirable that these specifications include bunch-stranded conductors not formed into rope-lay cables, in order to extend the bunch-stranded construction to the smallest sizes of flexible cables.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee B-1 reported results of the letter ballot vote as follows: Of a total membership of 30, 22 members returned their ballots, of whom 19 voted affirmatively, 1 negatively, and 2 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 1059.

Discussion has brought out the need for some further work on these specifications, incorporating certain suggestions received relative to the draft heretofore proposed for presentation to the Society. Upon completion of these revisions, and with a favorable vote by the committee, these specifications will be presented to the Society during the coming year looking toward their publication as tentative or perhaps for publicaiton as information and guidance in the ASTM BULLETIN.

Subcommittee X (C. S. Gordon, chairman) is studying specifications for lead or lead-alloy coated wires and has sent out a questionnaire on the subject to the committee.

Subcommittee XI (R. S. Pratt, chairman) has recommended that Committee B-2 on Non-Ferrous Metals and Alloys be requested to consider the preparation of specifications for non-oxygen-bearing copper, to which reference may be made in the specifications for copper wire.

Subcommittee XII (Alfred Bellis, chairman) is studying the desirability of preparing specifications for tinned-coated hard-drawn and medium-hard-drawn copper wire, and has sent out an inquiry to the committee covering this subject.

Subcommittee XIII (W. H. Bassett, Jr., chairman) has investigated the subject of bend tests and bend testing devices, including the R.C.A. bend tester, the Bell Telephone Laboratories wire tester, the Belden wire tester, and the Morehouse tester. Conclusions have not yet been reached, and the committee is continuing its work.

Subcommittee XIV (Alfred Bellis, chairman) has been studying the adequacy of the existing specifications for copper wire bars (B 4 – 27 and B 5 – 27) as a basis for requirements for some varieties and sizes of copper wire for electrical conductors.

There has been considerable discussion concerning the method of continuing this work and it has been decided to discontinue this subcommittee. It is felt that greater efficiency in outlining the needs of the industry and developing a solution may be obtained from a smaller group composed of two members from Committee B-1 and two from Committee B-2 on Non-Ferrous Metals and Alloys. This method of working out the problem will be submitted to Committee B-2 for consideration.

Subcommittee XV (G. E. Rolston, chairman) has been formed to study dimensional tolerances prescribed in the various specifications under the jurisdiction of Committee B-1. It has been brought to the attention of the committee that there are possible inconsistencies in the dimensional tolerances of similar specifications, and it also has been indicated that the dimensional tolerances for cables as prescribed in Specifications B 8 – 39 are not the same as those prescribed in the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 39 T), which are under the jurisdiction of Committee D-11 on Rubber Products. Recommendations by this subcommittee relative to revisions in the permissible variations in diameter prescribed in the Standard Specifications for Hard-Drawn Copper Wire (B 1 – 39) and for Medium-Hard-Drawn Copper Wire (B 2 – 39) are given later in this report.

Committee B-1 will endeavor to make recommendations on area tolerances which will be acceptable to the industry and which will make the specifications more generally applicable without modification.

I. REVISION OF TENTATIVE STANDARD

Tentative Specifications for Soft Rectangular and Square Copper Wire for Electrical Conductors (B 48 – 39 T):[2]

The committee recommends that these specifications be revised as follows and continued as tentative:

Classes.—In Sections 1 (*b*) and (*c*), 4 (*a*) and (*d*), and 5 (*a*) and (*c*) change the word "class" to read "type."

Section 4.—Change Paragraph (*c*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*c*) Retests.—If upon testing a sample from any coil or reel of wire, the results do not conform to the requirements [specified] ***prescribed*** in Paragraph*s* (a) ***and*** (*b*), two additional samples shall be tested, [and the average of the three tests shall determine the acceptance or rejection of the coil or reel] ***each of which shall conform to the prescribed requirements.***

Change Paragraph (*d*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*d*) ***For*** type B wire ***the tension test will not be required*** [need not conform to the tensile properties specified in Paragraph (a)].

Section 5.—Add two new paragraphs to this section, designated Paragraphs (*c*) and (*d*), to read as follows:

> (*c*) Bend tests shall be made upon representative samples by means of a suitable device and under the following conditions:
>
> The speed of bending shall be uniform and the bend shall be completed in not less than 3 nor more than 7 sec. The sample shall remain in its original plane and the inner side of the bent portion shall remain in contact with the mandrel, which shall not gouge the sample. At the conclusion of the test the sample shall be in contact with the mandrel through the entire angle of 180 deg., and the straight portions of the sample shall be parallel, the distance between them being equal to the diameter of the mandrel.
>
> (*d*) ***Retests.***—If upon testing a sample from any coil or reel of wire, the results do not conform to the requirements prescribed in Paragraphs (*a*), (*b*), and (*c*), two additional samples shall be tested, each of which shall conform to the prescribed requirements.

Reletter Paragraph (*c*) as (*e*) and change to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*e*) ***For*** type A wire ***the bend test will not be required*** [need not conform to the bending properties specified in Paragraph (a)].

Section 7 (*d*).—Change from its present form: namely,

> (*d*) The corners of the wire shall be rounded to a quarter circle of the radius, as determined by a radius gage, prescribed in the following table:

Specified Thickness, in.	0.751 in and over in Width	0.750 in. and under in Width
0.689 and over......	0.188 in.	0.188 in.
0.688 to 0.439......	0.125 in.	0.094 in.
0.438 to 0.226......	0.094 in.	0.063 in.
0.225 to 0.166......	0.063 in.	0.047 in.
0.165 to 0.126......	0.063 in.	0.031 in.
0.125 to 0.106......	half-round	0.031 in.
0.105 to 0.051......	half-round	0.025 in.
0.050 and under[a]...	half-round	half-round

[a] Square wire in this thickness range shall have the corners broken slightly.

to read as follows:

> (*d*) The corners of the wire shall be rounded to a quarter circle of the radius, as determined by a radius gage, prescribed in the following table:

Specified Thickness, in.	0.751 in. and over in Width	0.750 in. and under in Width
0.689 and over..	3/16 in.	3/16 in.
0.688 to 0.439...	1/8 in.	3/32 in.
0.438 to 0.226...	3/32 in.	1/16 in.
0.225 to 0.166...	1/16 in.	3/64 in.
0.165 to 0.126...	1/16 in.	1/32 in.
0.125 to 0.063[a]..	rounded edge	1/32 in.
0.062 and under.	rounded edge	rounded edge

[a] Square wire 0.102 in. and under shall have corner radii of 1/64 in.

Section 10 (*c*).—Change from its present form: namely,

> (*c*) Unless otherwise agreed upon, the wire shall be shipped in continuous lengths weighing not less than 135 lb.

to read as follows:

> (*c*) Unless otherwise agreed upon, the wire shall be shipped in continuous lengths of the following minimum weights:

Nominal Area, sq. mils	Minimum Weight, lb.
5001 and over	135
5000 to 2000	65
1999 and under	30

II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Standard Specifications for Hard-Drawn Copper Wire (B 1 – 39; ASA H4.2–1940) and Standard Specifications for Medium - Hard - Drawn Copper Wire (B 2 – 39; ASA H4.3–1940):[3]

At present Section 5 (*b*) of both of these specifications reads as follows:

> (*b*) The wire shall not vary from the specified diameter by more than the following amounts:

Diameter of Wire, in.	Permissible Variations in Diameter, plus or minus
Under 0.100	0.001 in. (1 mil)
0.100 or over	1 per cent

The corresponding section in the Standard Specifications for Soft or Annealed Copper Wire (B 3 – 39) is identical except that the value of permissible variation in diameter of 1 per cent applies to wire of diameters as small as 0.0100 in. and permissible variations in diameter for wire under 0.0100 in. is plus or minus 0.0001 in. (0.1 mil.).

The committee finds no valid reason for this discrepancy and therefore recommends that Section 5 (*b*) in Standard Specifications B 1 – 39 and B 2 – 39 be changed to read as follows:

> (*b*) Within the range of diameters included in Table I the wire shall not vary from the specified diameter by more than plus or minus 1 per cent.

III. ADOPTION OF TENTATIVE STANDARD AS STANDARD

Tentative Specifications for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (B 116 – 39 T):[4]

These specifications have been published as tentative for one year, and the committee upon inquiry finds no appreciable difficulty in meeting the prescribed requirements nor any suggestions as to revision. It is accordingly recommended that these specifications be approved for reference to letter ballot of the Society for adoption as standard.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE STANDARD			
Spec. for Soft Rectangular and Square Copper Wire for Electrical Conductors (B 48 – 39 T)	21	1	8
II. REVISIONS OF STANDARDS			
Spec. for Hard-Drawn Copper Wire (B 1 – 39), immediate adoption	28	1	2
Spec. for Medium-Hard-Drawn Copper Wire (B 2 – 39), immediate adoption	28	1	2
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Spec. for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (B 116 – 39 T)	19	0	11

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 34 members; 30 members returned their ballots, the results being shown in Table I.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 34 members; 30 members have returned their ballots, of whom 28 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

G. E. DEAN,
Secretary.

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[3] 1939 Book of A.S.T.M. Standards, Part I, pp. 551 and 555.

[4] *Ibid.*, p. 1063.

REPORT OF COMMITTEE B-2

ON

NON-FERROUS METALS AND ALLOYS

Committee B-2 on Non-Ferrous Metals and Alloys held two meetings during the year: on June 30, 1939, in Atlantic City, N. J., during the annual meeting of the Society, and in Detroit, Mich., on March 7, 1940. Also, the advisory committee met in Chicago, Ill., on October 25, 1939.

At the June, 1939, meeting E. E. Thum was elected chairman of Committee B-2 and C. E. Swartz was elected secretary.

Subsequent to the 1939 annual meeting Committee B-2 presented to the Society through Committee E-10 on Standards new Tentative Specifications for Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 – 39 T). These specifications were accepted[1] by Committee E-10 on August 24, 1939, and appear in the 1939 Book of A.S.T.M. Standards.[2]

I. New Tentative Standard

On the recommendation of Subcommittee II the committee presents for publication new Tentative Specifications for Pig Lead[3] to replace immediately the present Standard Specifications for Pig Lead (B 29 – 35). This recommendation was submitted to letter ballot of the committee which consists of 108 members; 55 members returned their ballots, of whom 38 voted affirmatively, 0 negatively, and 17 members marked their ballots "not voting."

II. Adoption of Tentative Standard as Standard

The committee recommends, as proposed by Subcommittee VI, that the Tentative Specifications for Lead-Coated Copper Sheets (B 101 – 35 T)[4] be approved for reference to letter ballot of the Society for adoption as standard. This recommendation was submitted to letter ballot of the committee which consists of 108 members; 68 members returned their ballots, of whom 42 voted affirmatively, 0 negatively, and 26 members marked their ballots "not voting."

Activities of Subcommittees

Subcommittee II on Refined Lead, Tin, Antimony, and Bismuth (T. A. Wright, chairman) has been working for some time on a revision of the Standard Specifications for Pig Lead (B 29 – 35). It was intended that the revised specifications would be submitted to the Society through Committee E-10 on Standards, and the specifications were considered by Committee E-10 at a meeting on August 24, 1939, and again on January 19, 1940 (at the latter meeting representatives of those in favor of and those opposed to the proposed revisions being present), but the specifications failed of approval, the difficulty lying in the proposed method of arrangement and designation of the several types of lead.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee B-2 reported results of the letter ballot vote as follows: Of a total membership of 102, 81 members returned their ballots, of whom 39 voted affirmatively, 2 negatively, and 40 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 1134.

[3] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 438.

[4] 1939 Book of A.S.T.M. Standards, Part I, p. 1109.

Subcommittee II was instructed to make a further effort to draft specifications that could be agreed to by all parties at interest, and at a meeting of the subcommittee held on February 16, 1940, a revised draft of the specifications was approved for reference to letter ballot of the subcommittee. The vote on this recommendation resulted as follows: From a subcommittee membership of 42, 27 members returned their ballots, of whom 18 voted affirmatively, 6 negatively, and 3 members marked their ballots "not voting." Objections to this draft of the specifications appeared to be based on the use of reference numerals in Table I on chemical requirements and to explanatory descriptive material being included. These objections were restated at the March, 1940, meeting of Committee B-2, at which meeting the specifications now being presented for publication as tentative,[3] were approved for reference to letter ballot of Committee B-2. The result of this vote is given earlier in this report. It was also voted to submit to letter ballot the addition to Table I of the following footnote:

> Corroding lead is a designation that has been used for many years in the trade to describe lead which has been refined to a high degree of purity.

The vote with respect to the addition of this footnote resulted as follows: From a membership of 108, 54 members returned their ballots, of whom 33 voted affirmatively, 0 negatively, and 21 members marked their ballots "not voting." Three affirmative ballots were received (from members whose business connections are closely affiliated) suggesting the desirability of making certain editorial changes.

Subcommittee III on White Metal Alloys (G. H. Clamer, chairman) has endeavored to reach agreement on the proposed revision of the Standard Specifications for Solder Metal (B 32 - 21) but it has found that the matter rests on doubts as to the necessity or practicability of establishing certain chemical limits. In view of this situation it will be necessary for the subcommittee to determine the actual facts before presenting the revised specifications for publication as tentative.[5]

Subcommittee IV on Refined Zinc and Wrought Zinc (E. H. Bunce, chairman), in recommending at the last annual meeting[6] that the Tentative Specifications for Rolled Zinc (B 69 - 37 T) be adopted as standard to replace the Standard Specifications B 69 - 29, also made the following editorial changes in the appendix to the specifications, which are given here as a matter of record:

> (*1*) In the temper values in the table on dynamic ductility and temper the $\frac{1}{2}$ per cent values were dropped since the limit of accuracy of the machine or use of the values is not closer than to the nearest 1 per cent.
>
> (*2*) The dynamic ductility values in the table were altered to the nearest 0 or 5 in the third decimal place, since here again this represents the limit of accuracy of the machine or use of the values.
>
> (*3*) A table on typical compositions of rolled zinc, quoted in part from the A.S.M. Metals Handbook, was added as being at least as valuable, particularly to those not entirely familiar with rolled zinc, as the table of physical properties.

Specifications B 69 - 39 were adopted as standard with the above editorial changes and appear in the 1939 Book of A.S.T.M. Standards.[7]

Subcommittee VI on Coated Metals (W. G. Schneider, chairman) is recommending, as mentioned earlier in this report, that the Tentative Specifications for Lead-Coated Copper Sheets (B 101 - 35 T) be adopted as standard. The vote on this recommendation in the subcommittee resulted as follows: From a membership of 34, 28 members returned their ballots, of whom 11 voted affirmatively, 0 negatively, and 17 members marked their ballots "not voting."

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[5] See Editorial Note, p. 168.

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 243 (1939).

[7] 1939 Book of A.S.T.M. Standards, Part I, p. 682.

At the June, 1940, meeting the present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 108 members; 68 members returned their ballots, of whom 62 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. E. THUM,
Chairman.

C. E. SWARTZ,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee B-2 presented to the Society through Committee E-10 on Standards new Tentative Specifications for Soft Solder Metal as a revision of and to replace immediately the Standard Specifications for Solder Metal (B 32 – 21). This recommendation was accepted by Committee E-10 on August 28, 1940, and the new tentative specifications appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 442.

REPORT OF COMMITTEE B-3

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held two meetings during the year: one in Atlantic City, N. J., on June 27, 1939, and one in Detroit, Mich., on March 6, 1940.

The committee has been active during the past year through its Subcommittees I on Total Immersion Test, II on Alternate Immersion Test, III on Spray Test, IV on Specifications, VI on Atmospheric Corrosion, and VIII on Galvanic and Electrolytic Corrosion in carrying forward the programs under the sponsorship of these subcommittees.

Activities of Subcommittees

Subcommittee I on Total Immersion Test (J. R. Freeman, Jr., chairman).—This subcommittee has revised the test program outlined in the 1939 report[1] and will conduct total immersion tests on commercial copper, phosphorized, in aerated and unaerated normal solutions of sulfuric acid, sodium chloride, and sodium hydroxide at 35 C. under closely controlled test conditions. Five cooperating laboratories will carry out the tests. It is hoped that this work will lead to the formulation of a recommended practice for total immersion testing.

Subcommittee II on Alternate Immersion Test (D. K. Crampton, chairman).—This subcommittee is preparing suggested methods of alternate immersion testing. It is felt in general that such test methods should avoid strict detail as to apparatus. A wide latitude in the size and shape of specimen is planned. The procedures are being so written as to permit using two methods: (*1*) a method in which complete drying of the specimens is forced as by a blast of hot air, and (*2*) a method in which the specimens are kept moist throughout the cycle.

The methods are being rewritten in accordance with the various suggestions received to date and will then be submitted for publication as tentative in order to elicit further comment. Following the publication of the methods, cooperative testing will probably be undertaken by the committee to obtain information on the merits and limitations of the test.

Subcommittee III on Spray Test (E. H. Dix, Jr., chairman).—Subcommittee III, having developed the Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 - 39 T), has been relatively inactive during the past year. The subcommittee met in Detroit, Mich., on March 6, 1940, at which time written comments on the tentative method were reviewed, and it was decided that further time should be allowed for the receipt of additional comments before any changes in the tentative method are made. It is therefore recommended that Method B 117 - 39 T be continued as tentative without revision until 1941.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 245 (1939).

Subcommittee IV on Specifications (E. A. Anderson, chairman).—Subcommittee IV held one meeting during the year, in Detroit, Mich., on March 6, 1940, in conjunction with the Joint Committee on Exposure Tests of Plating on Non-Ferrous Metals and Subcommittee VI on Finishing Properties of Die Castings of Committee B-6. Proposed Specifications for Electrodeposited Coatings of Nickel and Chromium on Copper and Copper Alloys, and on Zinc and Zinc Alloys have been prepared and were the subject of discussion at the March meeting. As soon as decision is reached on the sampling provisions, the specifications will be presented to the Society for publication as tentative.

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—Atmospheric corrosion tests on nonferrous metals and their alloys are being continued at the various test locations. The subcommittee does not contemplate at the present time removing any test samples from the racks for test for at least another three or four years.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (L. J. Gorman, chairman).—Committee B-3, on the recommendation of Subcommittee VIII, is sponsoring a paper on "Some Observations of the Potentials of Metals and Alloys in Sea Water," by F. L. LaQue and G. L. Cox, and a paper on "Controlling Factors in Galvanic Corrosion," by W. A. Wesley, which appear as Appendices I and II.[2] Further action on the sea water test is being deferred until these papers have been presented and studied.

The subcommittee is proceeding with plans for tests on stainless steels coupled with other metals in atmospheric exposures. In the present plan it is proposed to test 18 per cent chromium, 8 per cent nickel alloy steel coupled with the following metals and alloys:

Aluminum 2S,
Aluminum alloy 53S,
Copper (phosphorus deoxidized),
Iron (mild steel),
Lead (chemical lead),
Nickel,
Architectural bronze (brass) (56 Cu, 3.5 Pb, balance Zn), and
Zinc.

The exposure locations tentatively selected are as follows:

New York, N. Y.,
Altoona, Pa.,
Sandy Hook, N. J.,
State College, Pa.,
La Jolla, Calif., and
Key West, Fla.

It has been decided not to include Phoenix, Ariz., Rochester, N. Y., and Pittsburgh, Pa., as test sites, so as to reduce the number of specimens and the work involved in setting up the tests and analyzing the results. There is still some question as to the type of test specimen and the assembly of the specimens.

Attention is called to the 1939 report of this subcommittee covering results of atmospheric exposure tests on galvanic couples over a 7-yr. period.[3] The proposed tests on stainless steel will supplement this former work.

Since the meeting in Atlantic City, N. J., in June, 1939, the subcommittee has completed its preliminary work on the tests in sea water and on the exposure tests of stainless steels. It does not seem desirable to publish the results of these tests in the report of Committee B-3 because of their exploratory nature.

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[2] See pp. 670 and 690.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 247 (1939).

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Sam Tour.
Vice-Chairman, H. S. Rawdon.
Secretary, A. W. Tracy.

This report has been submitted to letter ballot of the committee which consists of 60 members; 45 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

T. S. Fuller,
Chairman.

Sam Tour,
Secretary.

REPORT OF COMMITTEE B-4

ON

ELECTRICAL-HEATING, ELECTRICAL-RESISTANCE, AND ELECTRIC-FURNACE ALLOYS

Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys held two meetings during the year: in Washington, D. C., on November 9 and 10, 1939, and in New York City on February 12 and 13, 1940, in addition to the meeting held during the annual meeting of the Society.

The committee is cooperating with the Joint A.S.T.M.-A.S.M.E. Research Committee on Effect of Temperature on the Properties of Metals, in the study of the behavior of metals at the higher temperatures.

In 1941 it is planned to publish a compilation of all the standards under the jurisdiction of Committee B-4.

The committee consists of 44 members, of whom 21 are classified as producers, 19 as consumers, and 7 as general interests.

I. NEW TENTATIVE STANDARD

The committee recommends that the proposed Tentative Methods of Testing Sleeves and Tubing for Radio Tube Cathodes be accepted for publication as tentative.[1]

II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following two tentative standards, with minor revisions, be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Method of Test for Flexivity of Thermostat Metals (*B 106 – 39 T*),[2] revised as follows:

Title.—Change from its present form to read: "Methods of Testing Thermostat Metals."

Section 1.—Add a new section on scope to read as follows:

> 1. These methods cover the procedures for testing thermostat metals.

Renumber present Sections 1 to 7 as Sections 2 to 8 and add a new heading "Flexivity" preceding new Section 2.

In Paragraph (*a*) change the minimum thickness of flat strips to which the method is applicable from "0.025 in." to read "0.015 in."

Change Paragraph (*b*) to read as follows by the addition of the italicized word and the omission of those in brackets:

> (*b*) The characteristics of thermostat metal are determined as a whole [when tested in the form of a flat strip], no account [is] *being* taken of the characteristics of the individual components.

Section 3.—Change from its present form to read as follows:

> 3. *Apparatus.*—The apparatus shall consist of the following:
>
> (*a*) *Specimen Carrier.*—A specimen carrier provided with two conical or knife-edge supports for locating the specimen. The test length,

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[1] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 448.

[2] 1939 Book of A. S. T. M. Standards, Part I, p. 1164.

that is the distance between the point of contact of the specimen with one support and the point of contact between the specimen and the other support, shall be known to within plus or minus 0.2 per cent and the line or plane passing through the points of contact shall be horizontal. The specimen carrier and supports shall hold the specimen without constraint so that the curvature due to its deflection will follow a vertical plane passing through the line joining the points of contact between specimen and supports.

(*b*) *Micrometer.*—A micrometer, traveling microscope, or equivalent device so connected to the specimen carrier that expansion during heating of the carrier or connecting parts will not cause appreciable displacement of the measuring device with respect to the supports.

(*c*) *Bath.*—A stirred liquid bath or uniformly heated enclosure, in which the specimen carrier together with adjustable electric heating source is placed, by means of which the specimen can be maintained at the desired temperatures, with a variation in temperature throughout the gage length of the specimen not to exceed 0.5 per cent of the temperature range used in the test.

(*d*) *Temperature Measuring Apparatus.*—Apparatus for making temperature measurements shall be of such accuracy that the interval between the two temperatures between which flexivity is to be measured, $T_2 - T_1$ in Eq. 1, will be known to plus or minus 0.5 per cent.

(*e*) *Deflection Index.*—Means shall be provided for measuring the deflection of the specimen at a point midway between the points of support and along the vertical line intersecting the line joining the points of support. Such means may comprise a fused quartz transmission rod disposed with its axis vertical and terminating in a point or knife-edge which shall engage the specimen midway between the points of contact with the supports. Such transmission rod shall be mounted in such manner that it is free to move in the direction of its axis. The rod shall bear at its free end an index suitable for microscopic observation, or else an electrical contact which with a micrometer will permit the changes of the deflection of the specimen to be accurately observed. Alternatively, the deflection of the mid point of the specimen may be directly observed by optical means whose line of sight is horizontal and passes through the vertical line through the mid point of the specimen. A micrometer screw with extended spindle making direct contact with the specimen may be used. In this case electrical means shall be provided that will indicate contact without significant disturbance of the specimen.

The measurement of the position of the midpoint of the test specimen shall be of such accuracy that the difference in position from temperature T_1 to temperature T_2 will be known to plus or minus 0.5 per cent of the maximum deflection for a given test.

(*f*) If a transmission rod is used, it and any attached parts shall be of such weight or so counterweighted that they will not cause a deflection greater than 1 per cent of the maximum to be produced by the action of the thermostat metal alone. When free, the thermostat metal assumes very nearly a circular curvature. Concentrated loading at the center of the specimen will cause the curvature to be other than circular, and may cause significant errors in the evaluation of flexivity. The location of the line passing through the points of contact between specimen and supports shall be known, with reference to the scale of the micrometer, to within plus or minus 0.002 in.

Section 4.—Change the last sentence of Paragraph (*d*) from its present form: namely,

> The ratio of the distance between supports to the thickness shall be determined to an accuracy of 0.10 per cent.

to read as follows:

> The distance between supports shall be known to an accuracy of 0.25 per cent.

Change Paragraph (*e*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*e*) [Dimensions] *The thickness* of *the* specimen shall be *accurately* determined [to an accuracy of 1 per cent] *within plus or minus 0.0001 in.* (*Note 1*) by means of a screw micrometer or equivalent method.
>
> *Note 1.—This corresponds to a permissible variation of plus or minus 0.66 per cent for specimens 0.015 in. in thickness, and plus or minus 0.2 per cent for specimens 0.050 in. in thickness.*

Number the present note following Paragraph (*e*) as Note 2 and change the first sentence of the second paragraph to read as follows by the addition of the italicized word and the omission of the words and figures in brackets:

> [In the case of very thin specimens (less than 0.010 in. in thickness)] The average thickness may *also* be calculated from measurements of length, average width, weight, and density.

Section 5 (a).—Change the second sentence of this paragraph to read as follows by the addition of the italicized word and the omission of the words in brackets:

> An amount of material extending a distance not less than [three times] *twice* the thickness shall be removed along each edge of the specimen, to eliminate material which may have been damaged in cutting.

Section 6 (h).—Add a new note to read as follows:

> NOTE.—The cumulative errors in the measurement of length, temperature, thickness, and deflection can produce discrepancies between successive flexivity determinations on the same strip as great as plus or minus 2.5 per cent. The probable error is substantially less than this. The test shall be made on at least two specimens from a given lot of material. If the values so obtained differ from their mean value by more than plus or minus 1.5 per cent of their mean value, these values should be rejected and the test repeated.

New Sections.—Add new Sections 9 and 10 on "Electrical Resistivity" and "Change of Resistance with Temperature" to read as follows:

> 9. Electrical resistivity shall be determined in accordance with the Standard Method of Test for Resistivity of Metallic Materials (A.S.T.M. Designation: B 63) of the American Society for Testing Materials.
>
> 10. Change of resistance with temperature shall be determined in accordance with the Standard Method of Test for Change of Resistance with Temperature of Metallic Materials for Electrical Heating (A.S.T.M. Designation: B 70) of the American Society for Testing Materials.

Tentative Method for Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (B 113 - 38 T),[3] revised as follows:[4]

Section 1.—Change the second sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> It is intended for [use on] *testing round* wire from approximately 0.010 to 0.117 in. (0.25 to 3.0 mm.) in diameter, *and rectangular and flattened wire not over 0.117 in. (3.0 mm.) in thickness where the ratio of width to thickness does not exceed 10.*

Change the footnote referring to the second sentence from its present form to read as follows:

> It has been observed that the bending moment obtained under method C (outlined in the Appendix to the 1936 Report of Committee B-4, see *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 166 (1936)) may be approximately the same as the initial bending moment obtained under this method for similar specimens, using the same test length and the same angle of deflection when the latter is in the neighborhood of 30 deg.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 44 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Testing Sleeves and Tubing for Radio Tube Cathodes....	31	1	3
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Flexivity of Thermostat Metals (B 106 - 39 T)...	29	0	5
Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (B 113 - 38 T),[a] as revised	28	1	6

[a] The recommendation for adoption as standard of this tentative method failed of approval at the annual meeting of the Society, see Summary of Proceedings, p. 31.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Life Tests (J. W. Harsch, chairman).—A study of the effect of ceramics and cements upon the life of heating element materials in contact with them has been made during the year and is being continued. Comparative tests are being made on nickel-chromium wire using the accelerated life test (B 76 - 39) in comparison with a so-called constant temperature test in which the temperature is adjusted every 24 hr. to the starting temperature.

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[3] 1939 Book of A. S. T. M. Standards, Part I, p. 1156.

[4] The recommendation for adoption as standard of this tentative method failed of approval at the annual meeting of the Society, see Summary of Proceedings, p. 31.

Subcommittee II on Electrical Tests (P. H. Dike, chairman).—A survey of the industry has shown a need for standard methods of test for electrical contact metals, and assurance has been received of sufficient support to justify undertaking this work. A program of work has been prepared to cover contacts suited for currents in the range of 0.1 to 50 amp. Tests are being made with a contacting device in use by the Edison General Electric Appliance Co. and with a device being built by the Material Laboratory of the New York Navy Yard in accordance with plans submitted by the committee.

Subcommittee IV on Mechanical Tests (P. H. Brace, chairman).—As mentioned earlier in this report, the scope of the Tentative Method of Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (B 113 - 38 T) is being extended to include round, rectangular, and flattened wire where the ratio of width to thickness does not exceed 10, and a footnote has been added referring to the bend test method C, described in the 1936 report of the committee. With this revision the method is being recommended for adoption as standard.

A method of test for resilience of strip (determined by measuring the amount of spring-back when flexed) is being developed at the request of the radio industry. A test of this character is already in use.

Subcommittee V on Wrought and Cast Alloys for High-Temperature Use (R. D. Van Nordstrand, acting chairman).—A high-temperature bend test intended for use on bars is under consideration, employing a small electric furnace within which the bars are tested.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman).—As indicated earlier in this report, the Tentative Method of Test for Flexivity of Thermostat Metals (B 106 - 39 T) has been recommended for adoption as standard with minor revisions. The subcommittee is now preparing methods of test for hardness and for electrical resistance.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (A. L. Chapman, chairman).—On the recommendation of this subcommittee proposed Methods of Testing Sleeves and Tubing for Radio Tube Cathodes are being presented for publication as tentative, as referred to earlier in this report. These test methods have been prepared at the request of the Radio Manufacturers Association. An earlier draft of the methods was published as information in the 1938 report of the committee.[5]

Considerable work has been done by the subcommittee in testing the thickness of nickel plating on thin steel strip using the Magnegage, and good agreement has been found with results obtained by chemical analysis. Further tests are being made. Methods of test for stiffness, brittleness, and hot tensile strength of tungsten wire are under consideration. Also, three laboratories are working on high-temperature tests of filament materials for radio tubes.

Subcommittee IX on Methods of Test for Alloys in Controlled Atmospheres (P. H. Brace, chairman).—The study of the action of "combusted gas" atmospheres on nickel-chromium resistor alloys has been continued, with four laboratories cooperating. Questions remain as to the causes of the "penetrating" type of deterioration, that type which causes substantially complete internal oxidation and embrittlement without striking changes in external appearance. Two laboratories have succeeded in reproducing the effect, while two others have failed, after repeated attempts. The fuels used differed, but the analysis of the resultant atmospheres was substantially the same. The rate of movement of the atmosphere with reference to the surface of the specimen is now regarded as a possible controlling factor. Further work has been planned with the thought of segregating the effects of fuel, rate of movement of test atmosphere, and cyclic variation of its composition. There is some reason for thinking that sulfur may play a part.

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 220 (1938).

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 44 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

DEAN HARVEY,
Chairman.

F. E. BASH,
Secretary.

REPORT OF COMMITTEE B-5 ON COPPER AND COPPER ALLOYS, CAST AND WROUGHT

Committee B-5 on Copper and Copper Alloys held two meetings during the year: in Atlantic City, N. J., on June 29, 1939, and in Washington, D. C., on March 1, 1940, with an attendance at the two meetings of the committee and its subcommittees of 51 and 45, respectively. In addition to these two meetings, the various subcommittees held a series of meetings in New York, N. Y., on December 11 and 12, 1939, at which 40 members were present.

The following additions and changes in membership have been approved:

W. W. Edens, Ampco Metal, Inc.

L. M. Long, Bunting Brass and Bronze Co.

L. L. Stott, Beryllium Corporation of Pennsylvania.

H. R. Klein, Bohn Aluminum and Brass Co.

R. S. Chadbourne, Boston Edison Co.

E. B. Powell, Association of Edison Illuminating Cos.

L. S. Fletcher, U. S. Army Ordnance Dept., Frankford Arsenal.

E. C. Jeter, Ford Motor Co.

J. B. Abele, Philadelphia Electric Co.

W. L. Bowler, Pure Oil Co.

W. E. Caldwell, Consolidated Edison Company of New York.

B. J. Higgins, Linderme Tube Co.

F. M. Harris, Pacific Gas and Electric Co.

F. C. Fyke, Standard Oil Development Co.

G. H. LeFevre, U. S. Smelting, Refining, and Mining Co.

E. J. Edwards, American Locomotive Co.

H. Y. Bassett to replace O. Z. Klopsch, representing the Wolverine Tube Co.

J. J. Curran replacing F. H. Morehead, representing the Walworth Co.

H. L. Smith replacing William Romanoff, representing the Non-Ferrous Ingot Metal Institute, with the latter named as alternate.

P. L. Irwin replacing J. A. Hance representing the Baldwin Locomotive Works.

A. L. Boegehold to represent General Motors Corp., previously without a designated representative.

H. J. Stein, representing Allis-Chalmers Manufacturing Co., replacing R. S. MacPherran, deceased.

G. H. Tinker representing the New York, Chicago, and St. Louis Railway Co., resigned.

A. L. Gemeny representing the American Association of State Highway Officials, deceased.

At the present time the committee consists of 100 members, of whom 84 are voting members; 32 are classified as producers, 34 as consumers, and 18 as general interest members.

Subsequent to the 1939 annual meeting, Committee B-5 presented to the Society through Committee E-10 on Standards revisions of the following tentative specifications:

Tentative Specifications for:

Brass Sheet and Strip (B 36 - 38 T),

Copper-Silicon Alloy Plate and Sheet (B 96 - 36 T),

Leaded Brass Sheet and Strip (B 121 - 39 T), and

Leaded-Nickel-Brass and Leaded-Nickel-Bronze (Nickel-Silver) Alloys in Ingot Form for Sand Castings (B 123 - 39 T).

The first three recommendations were accepted[1] by Committee E-10 on August 24, 1939, and the fourth recommendation was accepted on October 27, 1939. The specifications in their revised form appear in the 1939 Book of A.S.T.M. Standards.[2]

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-5 reported the following results of the letter ballot vote of a total of 57 ballots returned from a committee membership of 74: B 36 - 38 T, affirmative 38, negative 0, ballots marked "not voting" 19; B 96 - 36 T, affirmative 39, negative 0, ballots marked "not voting" 18; B 121 - 39 T, affirmative 38, negative 0, ballots marked "not voting" 19; B 123 - 39 T, affirmative 32, negative 0, ballots marked "not voting" 25.

The revised Standard Specifications for Copper Water Tube (B 88 - 39) have been approved by the American Standards Association as American standard (ASA No. H23.1-1939), and the Tentative Specifications for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 39 T) have been approved as American tentative standard (ASA No. H7-1939). Likewise, the Tentative Classification of Cast Copper-Base Alloys (B 119 - 39 T) has been endorsed by the American Foundrymen's Association.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following new specifications be accepted for publication as tentative:[3]

Tentative Specifications for:

Cartridge Brass Cartridge Case Cups,
Gilding Metal Sheet and Strip,
Gilding Metal Bullet Jacket Cups,
Copper Rods, Bars, and Shapes,
Brass Wire,
Miscellaneous Brass Tubes, and
Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings.

II. REVISION OF TENTATIVE STANDARDS

The committee recommends revision of the following six tentative specifications, as given in detail below:

Tentative Specifications for Brass Sheet and Strip (B 36 - 39 T):[4]

Section 3.—Change the copper content of alloy No. 8 from "64.5 to 67.5 per cent" to read "64.0 to 67.5 per cent."

Add a footnote to the table referring to alloy No. 8 to read as follows:

> Material to be free from beta constituent when examined at 75×.

Table I.—Change the Rockwell superficial 30-T hardness values from their present form to read as shown in the accompanying Table I.

TABLE I.—ROCKWELL SUPERFICIAL 30-T HARDNESS VALUES.

Alloy	No. 1		No. 2		No. 3		No. 4		Nos. 5 and 6		Nos. 7 and 8	
Temper	Min.	Max	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Quarter hard	29	51	34	54	38	58	42	60	43	60	43	60
Half hard	43	56	50	61	54	64	56	66	56	68	54	66
Three-quarter hard	50	60	56	64	60	68	63	70	65	72	65	71
Hard	54	62	60	67	65	70	68	73	70	74	68	73
Extra hard	60	65	65	70	69	74	72	76	74	77	73	76
Spring	62	67	68	71	72	75	75	78	76	78	75	78
Extra spring	63	68	69	72	73	76	76	79	77	79	76	79

Tentative Specifications for Copper-Silicon Alloy Plate and Sheet (B 96 - 39 T):[5]

Section 8.—Add the following sentence to this section:

> For purposes of these specifications, types A and C shall be considered to weigh 0.309 lb. per cu. in.

Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 39 T):[6]

Table I.—Change the chemical requirements for admiralty metal and aluminum brass from their present form: namely,

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[2] 1939 Book of A.S.T.M. Standards, Part I, pp. 1096, 1128, 1103, 1074.

[3] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, pp. 425, 429, 434, 375, 407, 386, and 369, respectively.

[4] 1939 Book of A.S.T.M. Standards, Part I, p. 1096.

[5] *Ibid.*, p. 1128.

[6] *Ibid.*, p. 1092.

	Admiralty[b]	Aluminum Brass[b]
Copper, min., per cent[a]....	70.0	76.0
Tin, per cent..............	0.90 to 1.20	1.25 max.
Aluminum, per cent	....	1.75 to 2.50
Nickel, per cent...........	....	1.25 max.
Lead, max., per cent.......	0.075	0.075
Iron, max., per cent.......	0.06	0.06
Zinc, per cent.............	remainder	remainder

[a] Silver counting as copper.

[b] If agreed upon between the manufacturer and the purchaser, these alloys may contain a maximum of 0.15 per cent antimony, or 0.10 per cent arsenic, or 0.20 per cent phosphorus.

to read as shown in the accompanying Table II.

In the case of the chemical requirements for arsenical copper, change the copper content from "99.20 per cent, min." to read "99.45 per cent, min.," and change the arsenic content from "0.15 to 0.75 per cent" to read "0.15 to 0.50 per cent."

TABLE II.—CHEMICAL REQUIREMENTS.

Type	Copper,[a] per cent	Tin, per cent	Aluminum, per cent	Lead, max., per cent	Iron, max., per cent	Zinc, per cent	Arsenic, per cent	Antimony per cent	Phosphorus, per cent
				ADMIRALTY METAL					
A.........	70.0 to 73.0	0.90 to 1.20	.....	0.075	0.06	remainder	.....	.....	.....
B.........	70.0 to 73.0	0.90 to 1.20	.....	0.075	0.06	remainder	0.10 max.	.....	.....
C.........	70.0 to 73.0	0.90 to 1.20	.....	0.075	0.06	remainder	.....	0.10 max.	.....
D.........	70.0 to 73.0	0.90 to 1.20	.....	0.075	0.06	remainder	.....	.....	0.10 max.
				ALUMINUM BRASS[b]					
A.........	76.0 min.	.....	1.75 to 2.50	0.075	0.06	remainder	.....	.....	.....
B.........	76.0 min.	.....	1.75 to 2.50	0.075	0.06	remainder	0.10 max.	.....	.....
C.........	76.0 min.	.....	1.75 to 2.50	0.075	0.06	remainder	.....	0.10 max.	.....
D.........	76.0 min.	.....	1.75 to 2.50	0.075	0.06	remainder	.....	.....	0.10 max.

[a] Silver counting as copper.

[b] Total other impurities, 0.30 per cent, max.

Section 6 (a).—Change to read as follows by the addition of the italicized words and figures and the omission of the words in brackets:

> 6. (*a*) A microscopic examination of the annealed tubes selected for test, except those of Muntz metal, shall show sufficient annealing for complete recrystallization with [a] uniform grain size. *The grain size shall not be less than 0.010 mm. nor* [not] in excess of 0.045 mm. [in] average diameter. Muntz metal tubes shall have been so annealed that when magnified at 75 diameters a properly prepared specimen shall show uniform and complete recrystallization. This requirement does not apply to hard-drawn tubes, end annealed, in which case no microscopic examination is required.

Tentative Specifications for Brass Pipe, Standard Sizes (B 43 – 39 T):[7]

Section 4.—In the case of Muntz metal and high brass, eliminate the chemical requirement for tin which reads "0.15 per cent, max."

Change the chemical requirements for admiralty metal to bring them in line with the requirements for admiralty metal recommended above in Specifications B 111 – 39 T in so far as lead and iron are permitted, by changing the iron content from "0.07 per cent, max." to read "0.06 per cent, max.," and the lead content from "0.07 per cent, max." to read "0.075 per cent, max."

Revise the chemical requirements for red brass to agree with the corresponding Government specifications, by changing the copper content from "84.00 to 87.00 per cent" to read "83.00 to 86.00 per cent," the lead content from "0.07 per cent, max." to read "0.06 per cent, max.," and the iron content from "0.07 per cent, max." to read "0.05 per cent, max."

Section 7.—Change the length of the

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[7] 1939 Book of A.S.T.M. Standards, Part I, p. 1083.

test specimen for the mercurous nitrate test from "3 in." to read "6 in."

Length Tolerances.—Add a new Section 13 on length tolerances to read as follows, renumbering the subsequent sections accordingly:

> 13. (*a*) Lengths of round seamless pipe shall conform to the tolerances prescribed in Table II (accompanying Table III).
>
> (*b*) In the case of specified straight lengths with ends included, at least 75 per cent by weight shall be the length specified, 25 per cent by weight may be shorter, but no piece shall be less than 50 per cent of the length specified.
>
> (*c*) Material ordered to random mill lengths shall include pipe of any lengths between 7 and 14 ft., at the discretion of the manufacturer.

TABLE III.—TOLERANCES IN INCHES FROM SPECIFIED LENGTHS OF ROUND SEAMLESS PIPE.

NOTE.—All tolerances in this table are plus.

Outside Diameter, in.	Straight Lengths				
	6 in. and under	Over 6 in. to 2 ft.	Over 2 to 6 ft.	Over 6 to 14 ft.	Over 14 ft.
1 and under	1/32	1/16	3/32	1/4	1/2
Over 1 to 2	1/16	3/32	1/8	1/4	1/2
Over 2 to 4	1/16	3/32	1/8	1/2	1/2
Over 4	..	1/8	1/4	1/4	1/2

Tentative Classification of Cast Copper-Base Alloys (B 119 – 39 T):[8]

Change the description of leaded tin bronze from "lead over 0.5 per cent, under 8 per cent" to read "lead over 0.5 per cent, under 6 per cent."

Change the description of high-leaded tin bronze from "lead over 8 per cent" to read "lead over 6 per cent."

Change the description of leaded bronze from "lead over 20 per cent" to read "lead over 30 per cent."

Tentative Specifications for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 38 T):[9]

Table I.—In the case of class C castings, change the chemical requirement for "copper, 82 per cent, max." to read "copper plus tin plus lead plus nickel, 98.50 per cent, min." Change the lead content from "8 to 11 per cent" to read "9 to 11 per cent," and the phosphorus content from "0.7 to 1.0 per cent" to read "0.10 per cent, min." Add a requirement for nickel to read "1 per cent, max." and omit the requirement for other impurities which reads "0.5 per cent, max."

Table II.—Add physical properties for class C castings to read "maximum compression under 10,000 psi.....0.001 in."

Tentative Specifications for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 – 39 T):[10]

The revision being recommended in these specifications consists primarily of a rearrangement of the tabulated requirements which will make the specifications in their revised form[11] more useful to the purchaser.

III. REVISIONS OF STANDARDS

The committee recommends for immediate adoption, revisions in certain specifications, as indicated below, and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for Seamless Copper Boiler Tubes (B 13 – 33;[12] *A.S.M.E. Boiler Construction Code No. S-22):*

The following revisions are recommended in order to bring the requirements for arsenical copper in line with those proposed above in Specifications B 111 – 39 T:

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[8] 1939 Book of A.S.T.M. Standards Part I, p. 1077.

[9] *Ibid.*, p. 1071.

[10] *Ibid.*, p. 1118.

[11] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 411.

[12] 1939 Book of A.S.T.M. Standards, Part I, p. 632.

Section 3 (a).—Change from its present form: namely,

(*a*) *Arsenical Copper.*—Arsenical copper shall contain from 0.15 to 0.35 per cent of arsenic, and shall not contain more than 0.120 per cent of impurities, exclusive of arsenic and silver.

TABLE IV.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Cartridge Brass Cartridge Case Cups	28	0	23
Spec. for Gilding Metal Sheet and Strip	28	0	23
Spec. for Gilding Metal Bullet Jacket Cups	30	0	24
Spec. for Copper Rods, Bars, and Shapes	34	0	20
Spec. for Brass Wire	33	0	18
Spec. for Miscellaneous Brass Tubes	37	0	17
Spec. for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings	27	0	24
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Brass Sheet and Strip (B 36 – 39 T)	32	0	19
Spec. for Copper-Silicon Alloy Plate and Sheet (B 96 – 39 T)	31	0	20
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 39 T)	30	2	19
Spec. for Brass Pipe, Standard Sizes (B 43 – 39 T)	30	0	21
Classification of Cast Copper-Base Alloys (B 119 – 39 T)	27	0	24
Spec. for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 38 T)	27	0	24
Spec. for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 – 39 T)	31	0	20
III. REVISIONS OF STANDARDS			
Spec. for Seamless Copper Boiler Tubes (B 13 – 33), immediate adoption.	31	0	20
Spec. for Sheet Copper-Silicon Alloy (B 97 – 39), immediate adoption	28	0	23
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 39), immediate adoption	31	0	20
Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 – 39), immediate adoption	31	0	20
Spec. for Steam or Valve Bronze Castings (B 61 – 36), immediate adoption	28	0	23
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 – 38 T)	30	0	21
Spec. for Phosphor Bronze Sheet and Strip (B 103 – 39 T)	29	0	22
V. REVERSION OF STANDARDS TO TENTATIVE			
Spec. for Cartridge Brass (B 19 – 29), as revised	31	0	20
Spec. for Naval Brass Rods for Structural Purposes (B 21 – 29), as revised	35	0	19
Spec. for Seamless Copper Tubes (B 75 – 30), as revised	32	1	18
VI. WITHDRAWAL OF STANDARDS			
Spec. for Cartridge Brass Disks (B 20 – 29)	30	0	21
Spec. for Leaded-Nickel-Brass and Leaded-Nickel-Bronze (Nickel-Silver) Alloys in Ingot Form for Sand Castings (B 123 – 39 T)	28	0	23

to read as follows:

(*a*) *Arsenical Copper.*—Arsenical copper shall have a purity of at least 99.45 per cent as determined by electrolytic assay, silver being counted as copper, and the arsenic content shall be 0.15 to 0.50 per cent. The total of impurities shall not exceed 0.120 per cent, exclusive of arsenic and silver.

Standard Specifications for Sheet Copper-Silicon Alloy (B 97 – 39):[13]

Section 11.—Add a new sentence at the end of this section to read as follows:

For purposes of these specifications, types A and C shall be considered to weigh 0.309 lb. per cu. in., and type B 0.316 lb. per cu. in.

Standard Specifications for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 39), and for Copper-Silicon Alloy Wire for General Purposes (B 99 – 39):[14]

At the end of these specifications add an Explanatory Note to read as follows:

NOTE.—For purposes of these specifications, the density shall be taken as 0.309 lb. per cu. in. for types A and C, and 0.316 lb. per cu. in. for types B and D.

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[13] 1939 Book of A.S.T.M. Standards, Part I, p. 646.

[14] *Ibid.*, pp. 625, 650.

Standard Specifications for Steam or Valve Bronze Castings (B 61 – 36):[15]

Section 3.—Change the chemical requirement for lead from "1.5 to 2.0 per cent" to read "1.0 to 2.0 per cent."

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following two tentative specifications be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for:[16]

Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 – 38 T), and

Phosphor Bronze Sheet and Strip (B 103 – 39 T).

V. REVERSION OF STANDARDS TO TENTATIVE

The committee recommends that the following three standard specifications be reverted to tentative and revised:[17]

Standard Specifications for:

Cartridge Brass (B 19 – 29),

Naval Brass Rods for Structural Purposes (B 21 – 29), and

Seamless Copper Tubes (B 75 – 30).[18]

VI. WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the Standard Specifications for Cartridge Brass Disks (B 20 – 29),[19] and the Tentative Specifications for Leaded-Nickel-Brass and Leaded-Nickel-Bronze (Nickel-Silver) Alloys in Ingot Form for Sand Castings (B 123 – 39 T).[20]

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 83 voting members, with the results shown in Table IV.

ACTIVITIES OF SUBCOMMITTEES

In an effort to render the various specifications under the jurisdiction of Committee B-5 more useful to industry, the various subcommittees have been cooperating with Governmental bureaus, such as the War Department, Federal Specifications Executive Committee, the Navy Department, Bureau of Aeronautics, and other agencies, in an effort to correlate the requirements of A.S.T.M. specifications and the corresponding Governmental specifications. It is the thought of this committee that by such correlation industry may be better prepared to supply the raw materials required by these agencies in case of emergency. Furthermore, it is hoped that the various Governmental agencies will be apprised of the various technological advances made in industry and consequently that both sets of specifications will be so prepared as to reflect the latest and best commercial practices.

Subcommittee I on Copper-Zinc Sheet and Strip (G. H. Harnden, chairman) has prepared revised Specifications for Cartridge Brass Sheet, Strip, and Disks, and new Specifications for Cartridge Brass Cartridge Case Cups, for Gilding Metal Sheet and Strip, and for Gilding Metal Bullet Jacket Cups. A review of the uses in industry of the Standard Specifications for Cartridge Brass (B 19 – 29) and for Cartridge Brass Disks (B 20 – 29) indicated that radical revisions of these two specifications were desirable. As a result of cooperative efforts with the War Department, Specifications B 19 have been reverted to tentative and revised to include requirements for both cartridge brass sheet and disks. Accordingly, it is recommended that the present Specifications for Cartridge Brass Disks (B 20 – 29) be withdrawn since these requirements no longer cover the needs of industry, whereas revised Specifications B 19 will not only cover requirements for cartridge brass sheet and strip but also for cartridge brass disks. In addition, new specifications were prepared to cover the needs of industry and the War Department with respect to gilding metal, cartridge case cups, and bullet jacket cups.

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[15] 1939 Book of A.S.T.M. Standards, Part I, p. 600.

[16] *Ibid.*, pp. 1131, 1112.

[17] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, pp. 419, 380, 391.

[18] See Editorial Note, p. 186.

[19] 1939 Book of A.S.T.M. Standards, Part I, p. 659.

[20] *Ibid.*, p. 1074.

Subcommittee I has also recommended minor revisions in the Tentative Specifications for Brass Sheet and Strip (B 36 – 39 T) by changing the chemical composition of alloy No. 8 to agree with present commercial practice and modifying the Rockwell superficial hardness values of the 30-T scale to agree with present commercial limits. During the past year, a need has also arisen in industry to cover the manufacture of brass sheet and strip by hot-rolling practices. Accordingly, minor revisions in Specifications B 36 – 39 T have been undertaken to facilitate this practice.

Subcommittee II on Copper-Tin Sheet and Strip (R. J. Wheeler, chairman) has recommended the adoption as standard of the Tentative Specifications for Rolled Copper-Alloy Bearing and Expansion Plates for Bridges and Other Structural Uses (B 100 – 38 T), and for Phosphor Bronze Sheet and Strip (B 103 – 39 T).

Subcommittee III on Copper-Nickel-Zinc Sheet and Strip (E. S. Bunn, chairman) is investigating the effect of small amounts of aluminum and magnesium on the physical properties of copper-nickel-zinc and copper-nickel alloys. This will ultimately lead to revision of the chemical composition requirements in the Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 39 T) to insure higher quality of these materials through control of the aluminum content.

Subcommittee IV on Miscellaneous Copper-Base Sheet and Strip Alloys (L. A. Ward, chairman) has recommended a revision of the Tentative Specifications for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 – 39 T) which will make these specifications more useful to the purchasers of this type of material. This subcommittee has also recommended minor changes in the Standard Specifications for Sheet Copper-Silicon Alloy (B 97 – 39) and the Tentative Specifications for Copper-Silicon Alloy Plate and Sheet (B 96–39 T) to include density requirements for the various types of alloys covered by these specifications, to meet consumer demand for information of this type.

Subcommittee V on Miscellaneous Copper-Base Wire and Rod Alloys (W. H. Bassett, Jr., chairman) has recommended that the Standard Specifications for Naval Brass Rods for Structural Purposes (B 21 – 29) be reverted to tentative and revised to include two new leaded naval brass alloys being used extensively in industry. Minor revisions in the present alloy, which will be designated grade A, are recommended to meet the requirements of the Navy Department. The two new leaded alloys will be designated grades B and C, respectively. The subcommittee has also recommended for publication as tentative new Specifications for Copper Rods, Bars, and Shapes for general use, to meet the requirements of the Navy Department and industry for an alloy of this type, and Specifications for Brass Wire for which no specification requirements were available.

Minor revisions have been recommended in the Standard Specifications for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 39), and for Copper-

Silicon Alloy Wire for General Purposes (B 99 – 39) to include density requirements for the various alloys covered by these specifications. These changes are in line with those which have been recommended above by Subcommittee IV in Specifications B 96 – 39 T and B 97 – 39.

Subcommittee VI on Condenser Tubes (G. C. Holder, chairman) has recommended changes in the Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 39 T). These revisions consist essentially of the introduction of four grades of admiralty metal and four grades of aluminum brass condenser tubes, one of which contains no arsenic, antimony, or phosphorus, and three other grades, each of which permits the presence of small amounts of one of these elements, that is, arsenic, antimony, or phosphorus as an added ingredient. These changes are made to cover various types of admiralty metal and aluminum brass tubes which have found widespread usage in industry to meet specific engineering requirements. Other minor changes have been recommended to improve the quality of the materials covered by these specifications.

Subcommittee VII on Copper or Deoxidized Copper Tubes (H. Y. Bassett, chairman) has recommended that the Standard Specifications for Seamless Copper Tubes (B 75 – 30) be reverted to tentative and revised[18] to bring the specifications in line with present industrial requirements. It has also recommended minor revisions in the Standard Specifications for Seamless Copper Boiler Tubes (B 13 – 33). The latter changes are intended to bring the requirements of these specifications in line with the changes being made in the condenser tube specifications (B 111 – 39 T).

A revision of the Standard Specifications for Copper Water Tube (B 88–39) to limit light temper to types K and L tubes is now under consideration.

Subcommittee VIII on Copper Alloy Tubes for General Use (Alan Morris, chairman) recommends a revision of the composition of the various alloys listed in the Tentative Specifications for Brass Pipe, Standard Sizes (B 43 – 39 T), to effect better agreement with the requirements of other A.S.T.M. specifications, such as the condenser tube specifications (B 111 – 39 T). It is recommending, also, the publication as tentative of new Specifications for Miscellaneous Brass Tubes for the purpose of controlling these materials since no specification requirements have been available heretofore.

Subcommittee IX on Copper-Base Alloy Forgings (L. A. Ward, chairman) has under consideration minor revisions in the Tentative Specifications for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 39 T), which have been found desirable as a result of experience obtained from the use of the present engineering requirements.

Subcommittee X on Copper-Base Alloys for Sand Castings (G. H. Clamer, chairman) has recommended the withdrawal of the Tentative Specifications for Leaded-Nickel-Brass and Leaded-Nickel-Bronze (Nickel-Silver) Alloys in Ingot Form for Sand Castings (B 123 – 39 T) because requirements covering these alloys will be contained in the specifications for copper-base alloys in ingot form now in preparation. Minor revisions in the Tentative Classification of Cast Copper-Base Alloys (B 119 – 39 T) are also recommended. The subcommittee has presented for publication as tentative new Specifications for Leaded High-Strength Yellow Brass Castings.

Work is rapidly approaching completion on the new specifications for copper-base alloys in ingot form which will replace the Standard Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 36). It is anticipated that the new specifications will be presented to the Society through Committee E-10 on Standards subsequent to the annual meeting, in which case it will also be recommended that Specifications B 30 – 36 be withdrawn.[18]

Subcommittee X also recommends that the Tentative Specifications for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 38 T) be revised to include physical requirements as to maximum allowable set for class C alloy and slight modifications in the chemical composition of this same alloy to conform with present practice.

Subcommittee XI on Methods of Test for Copper and Copper Alloys (A. J. Phillips, chairman) is conducting a series of tests to permit standardization of the mercurous nitrate test for copper-base alloys and the pin or expansion tests specified in various tube specifications. It has also referred to Committee E-1 on Methods of Testing the problem of standardizing the method of making Rockwell hardness tests on rods and tubes.

Subcommittee XI has likewise discussed the determination of the yield point as prescribed in the various specifications under the jurisdiction of this committee. It is anticipated that a request for a change in the methods of specifying yield point will be made to Committee E-1 to insure uniformity in the various specifications covering non-ferrous metals.

Subcommittee XII on Publication of General Information (C. S. Cole, chairman) is preparing a paper on the classification of wrought copper and copper-base alloys. It is also reconsidering the possibility of having all the A.S.T.M. specifications pertaining to copper-base alloys published as a separate compilation.

A special committee, consisting of J. R. Freeman, Jr., and J. W. Bolton, is cooperating with the Joint A.S.M.E.-A.S.T.M. Research Committee on Effect of Temperature on the Properties of Metals in considering the effect of temperature on the non-ferrous alloys under the jurisdiction of Committee B-5. It is hoped that as a result of this cooperation, specific information will be made available on the effect of temperature on tensile strength and modulus of elasticity, with a recommendation with respect to the maximum temperature for continuous operation of the various materials.

The present officers of Committee B-5 have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 84 voting members; 51 members returned their ballots, of whom 47 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

C. H. GREENALL,
Chairman.

C. H. DAVIS,
Secretary.

(See Editorial Note, page 186.)

EDITORIAL NOTE

Subsequent to the annual meeting Committee B-5 presented to the Society through Committee E-10 on Standards new Tentative Specifications for Copper-Base Alloys in Ingot Form for Sand Castings as a revision of and to replace immediately the Standard Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 36); also a revision of the Tentative Specifications for Seamless Copper Tubes (B 75 – 40 T). These recommendations were accepted by Committee E-10 on August 28, 1940, and the new and revised tentative specifications appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, pp. 362 and 391.

REPORT OF COMMITTEE B-6

ON

DIE-CAST METALS AND ALLOYS

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: one in Atlantic City, N. J., on June 29, 1939, and one in Detroit, Mich., on March 6, 1940, with an attendance of 33 and 44, respectively, at the two meetings of the committee and its subcommittees. Certain of the subcommittees also held other meetings at various times during the year.

A balance of $185.42 is reported by Society headquarters in the fund of Committee B-6 as of December 31, 1939.

The committee has undertaken to raise $2000 from producers and consumers of die castings to support an extension of its present program of research. These funds are required to defray the expense of building exposure racks for the mounting of test specimens, transportation of these racks to the exposure locations, removal of test specimens from the racks and return to the testing laboratories as well as any expenses incidental to the cost of installing such racks on location. The various committee members contribute the test specimens required for these studies and make the necessary laboratory tests to determine initial physical properties and the physical properties after exposure. This program is an extension of the comprehensive investigations[1] carried out by Committee B-6 on 12 aluminum- and 10 zinc-base alloys over a 10-year period and of studies now under way on five lead- and tin-base die-casting alloys, three zinc-base and four magnesium-base alloys as well as three aluminum-base alloys of varying degrees of purity, as discussed later in this report under the activities of Subcommittees I, III, and V.

The committee is cooperating with a Joint Committee, composed of representatives of Committees A-5 onCorrosion of Iron and Steel, and B-3 on Corrosion of Non-Ferrous Metals and Alloys in the preparation of specifications for electrodeposited coatings on ferrous and non-ferrous alloys. As a result of this work proposed tentative specifications covering various electrodeposited coatings are being prepared for presentation to the Society.

Committee B-6 has under consideration the advisability of relinquishing jurisdiction in the matter of standards for electrodeposited coatings to a separate standing committee which it has been suggested be organized to handle such standards. This suggestion has been referred by the Executive Committee of the Society to the officers of the interested standing committees.

The advisory committee has recommended changes in the by-laws of Committee B-6 in those sections covering the attendance of members at meetings and the return of letter ballots.

A number of additions and changes in the membership of the committee have been approved during the year. At the present time the committee consists of 80 members, 67 of whom are voting members; 26 are classified as producers, 23 as consumers, and 18 as general interest members.

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[1] Report of Subcommittee V on Exposure and Corrosion Tests of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, pp. 265, 271 (1932), and Vol. 35, Part I, p. 190 (1935).

I. REVISION OF TENTATIVE STANDARD

The committee recommends that the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 - 39 T)[2] be revised as follows and continued as tentative:

Section 3.—In the table of chemical composition omit the requirements for alloys Nos. 12 and 13 which read "other ingredients (cadmium, tin) . . . 0.3 per cent, max."

Following the table add two notes to read as follows:

NOTE 1.—Analysis shall regularly be made only for the elements specifically mentioned in this table. If, however, the presence of other elements is suspected or indicated in the course of routine analysis, further analysis shall be made to determine that these other elements are not in excess of 0.3 per cent.

NOTE 2.—The following applies to all specified limits in this table: For purposes of acceptance and rejection an observed value or a calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit.

This recommendation has been submitted to letter ballot of the committee which consists of 67 voting members; 47 members returned their ballots, of whom 34 have voted affirmatively, 0 negatively, and 13 members marked their ballots "not voting."

II. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The committee recommends that the following tentative specifications be continued as tentative without revision:

Tentative Specifications for:

Aluminum-Base Alloy Die Castings (B 85 - 39 T),
Zinc-Base Alloy Die Castings (B 86 - 38 T), and
Lead- and Tin-Base Alloy Die Castings (B 102 - 39 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. E. Thum, chairman) has completed the 5-yr. tests on the three special aluminum-base alloys Nos. IVa, Va, and V. The results of these tests, compiled by E. H. Dix, Jr., are given in Appendix I.

The chairman reported that the problem of harmonizing the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 - 39 T) with the U. S. Government specification for aluminum die castings, which was considered at the June, 1939, meeting, has been solved in a very satisfactory manner, the subcommittee having received from the Procurement Division of the Government a revised copy of the federal specification in which the composition limits have been made to conform with those of the A.S.T.M. specifications. Minor changes have been made in the Government specification with respect to the chemical limits for certain special alloys of high purity required by some of the Arsenals, but the great bulk of the die castings will be bought under commercial composition requirements as contained in the A.S.T.M. specifications.

Subcommittee I is arranging with two producers for the die casting of test specimens of an alloy of 8 per cent magnesium, balance aluminum, to be designated alloy No. 101, one set to be made on a gooseneck type of machine, and the other on a plunger type. Bars are to be segregated hour by hour during the production of a full shift in order to study the effect of time on composition, that is, loss of magnesium and iron pickup. Tension and impact tests and chemical analyses will then be used to determine the selection of bars for further studies, including salt spray, outdoor exposure, and other tests. Nos. 102 to 112, inclusive, have been reserved for future aluminum-base alloys which may be studied by the subcommittee. The subcommittee is seeking wider coöperation from various testing laboratories in the conducting of salt spray tests.

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[2] 1939 Book of A.S.T.M. Standards, Part I, p. 1151.

Subcommittee II on Zinc-Base Die-Casting Alloys (C. T. Prendergast, chairman) has been reorganized. Because of ill health, C. E. Heussner, who for many years has ably served as chairman of this subcommittee, has felt it necessary to relinquish his duties. Subcommittee II has under consideration a proposed revision of the Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 38 T) with respect to the composition requirements for alloy No. XXV.

Subcommittee III on Tin- and Lead-Base Die-Casting Alloys (G. O. Hiers, chairman) is continuing its studies on the five lead- and tin-base die-casting alloys. The creep studies on these alloys have been started but no data will be available for approximately two years.

Subcommittee IV on Physical Tests of Die Castings (R. L. Templin, chairman) is considering impact and fatigue testing of die castings. The size and shape of the Charpy impact specimen are being reviewed and the type of specimen suitable for use in the impact-tension testing of die-cast specimens has been referred to this subcommittee for study.

Subcommittee V on Exposure and Corrosion Tests (W. M. Peirce, chairman) is continuing its studies of the atmospheric corrosion tests of die-casting alloys. As discussed earlier in this report, it plans to extend these studies provided the necessary funds are raised among the members of Committee B-6.

The 12 aluminum-base alloys Nos. I to XII are still being continued in the atmospheric corrosion tests after an exposure period of 10 years. The results of inspections and tests of these alloys after 1-year and 5-year exposures were included in the 1932 and 1935 reports.[1]

Specimens of the 10 zinc-base die-casting alloys which were also inspected and tested after exposures of 1 and 5 years[1] have now been exposed for 10 years at 6 outdoor and 4 indoor locations. Specimens have been removed at the end of this 10-year exposure period and the results of these tests are given in Appendix II.

The data include the results of tests for tensile strength, elongation, Charpy impact strength, Brinell hardness, and expansion of all the alloys except alloy No. XXI. In the case of alloy No. XXI, the specimens are being continued in the atmospheric corrosion tests but the expansion data on this alloy are given in Table II of Appendix II. None of the zinc-base alloys included in the tests are in commercial use today although alloy No. XXI with slight modifications is identical with that now covered in the Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 38 T). However, as a result of the investigations carried on by the subcommittee, the improvements in alloy No. XXI and the introduction of two new alloys, designated alloys Nos. XXIII and XXV, have resulted in widespread commercial usage of zinc-base die-casting alloys.

Exposure tests on three new zinc-base and four magnesium-base alloys die cast late in 1938 are now under way.

Subcommittee VI on Finishing Properties of Die Castings (J. C. Fox, chairman) is preparing a series of papers covering all the up-to-date methods of finishing various types of die-casting alloys. These papers will probably be presented to the Society at the 1941 annual meeting. This subcommmittee has also been cooperating with the Joint Committee, mentioned earlier in this report, in the preparation of specifications for electrodeposited coatings on ferrous and nonferrous alloys.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (J. A. Gann, chairman) has recommended a minor revision in the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 - 39 T), as mentioned earlier in this report, to conform with changes being recommended by Committee B-7 on Light Metals and Alloys[3] in the Tentative Specifications for Magnesium-Base Alloy Ingot for Remelting (B 93 - 39 T).

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 67 voting members; 44 members return their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

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[3] See p. 202.

APPENDIX I

AN INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS Nos. IVa, Va, AND V

The 1935 report[1] of Subcommittee I described the preparation of the specimens for this investigation and presented the results of chemical analyses and "original" mechanical properties tests. The following year the results of the accelerated corrosion tests were published,[1] with the general conclusion that, although the differences were not large, the resistance of alloy No. Va to severely corrosive conditions was superior to that of alloys Nos. IVa and V. Although it was planned to examine both visually and under the microscope specimens removed from the atmospheric exposures annually, these plans went awry and the first examination of this type was made only last year.[2]

The present report describes the examination and tests of specimens removed from the atmospheric exposure racks after an exposure of 5 yr. The results, as were expected, show relatively small differences between the several materials, largely because corrosion has not progressed to a significant degree.

Description:

Since Table I of the 1935 report[1] details the distribution of specimens in the tests, it will suffice here merely to say that three racks, each containing 180 round tension specimens and 60 flat tension specimens, were exposed at the Sandy Hook, N. J., New York, N. Y., and Altoona, Pa., test sites. In accordance with the practice established by Subcommittee V, the round specimens were exposed with the axis in a horizontal plane. The flat specimens, since they were to be employed for studying the rate of surface attack, were exposed so that one flat surface was horizontal.

At the end of 5-yr. exposure, 60 round tension specimens and 12 of the flat specimens were removed from each rack for test and examination. Since there are two lots of three alloys in the test, this provided 10 round and 2 flat specimens of each material. Five of the round specimens of each material were tested by the Hawthorne Plant of the Western Electric Co. while the other five were tested by the Aluminum Research Laboratories. Each laboratory tested five "blanks" of each material taken from a group stored indoors for 5 yr. As with the specimens from the accelerated corrosion tests (1936 report),[1] the mechanical property tests were made with Amsler hydraulic testing machines using the testing procedures regularly employed for non-ferrous specimens. The yield strengths reported were determined by the aid of an automatic, autographic electrical extensometer. The visual and microscopic examinations of the flat specimens were made by the Aluminum Research Laboratories; the latter, using polished and etched transverse sections and a magnification of 100×.

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[1] An Investigation of Aluminum Die-Casting Alloys Nos. IVa and Va, Appendix I, Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats, Vol. 35, Part I, p. 184 (1935); Vol. 36, Part I, p. 182 (1936).

[2] Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 278 (1939).

Test Results:

Tables I and II show the average mechanical properties of the "blanks" tested after 5-yr. storage indoors and the percentage change in properties for the specimens exposed at the three outdoor stations (as compared with the "blanks"). Figure 1 shows, in a different form, the tensile strength of the various specimens after 5-yr. storage or exposure. In Table III are given the results of the microscopic examination of the flat specimens. The results of the visual examination are incorporated in the discussion.

TABLE I.—INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS NOS. IVa AND Va. RESULTS OF EXPOSURE TO THE ATMOSPHERE FOR 5 YR.

Alloy[b]	Original Properties[a]			Sandy Hook, N. J.			New York, N. Y.			Altoona, Pa.		
				Percentage Change			Percentage Change			Percentage Change		
	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Tensile Strength	Yield Strength	Elongation in 2 in.	Tensile Strength	Yield Strength	Elongation in 2 in.	Tensile Strength	Yield Strength	Elongation in 2 in.
TESTED BY ALUMINUM RESEARCH LABORATORIES												
No. 14865 (IVa).....	27 425	10 500	6.3	−1.9	+1.9	−9.5	−3.9	0	−11.1	−2.3	−0.5	0
No. 15431 (IVa).....	26 540	12 650	5.2	−0.1	−0.4	+5.8	−2.6	−2.4	−1.9	−3.8	−1.2	0
No. 14866 (Va)......	33 530	16 600	3.0	−1.6	+1.2	−13.3	−5.9	+0.9	−40.0	−3.1	+0.9	−6.7
No. 15432 (Va)......	29 820	17 500	2.3	−4.8	−1.4	−17.4	−16.1	−1.1	−34.8	−9.5	+3.4	−34.8
No. 14867 (V).......	35 370	19 500	2.5	−0.7	+1.3	−4.0	−1.4	+0.3	−16.0	−0.7	−1.8	−20.0
No. 15433 (V).......	30 140	20 350	1.8	−9.3	−4.7	−16.7	−3.8	−4.9	−11.1	−5.6	−3.9	−44.4
TESTED BY WESTERN ELECTRIC CO.												
No. 14865 (IVa).....	27 980	....	5.5	−3.6	....	−1.8	−2.4	....	−7.3	−3.7	....	+3.6
No. 15431 (IVa).....	27 700	....	5.3	−5.6	....	−13.2	−4.8	....	−11.3	−7.4	....	−20.8
No. 14866 (Va)......	34 280	....	2.5	−4.7	....	−4.0	−1.1[c]	....	0	−5.5	....	0
No. 15432 (Va)......	28 420	....	1.7	−2.0	....	+11.7	−4.8	....	−11.7	−2.0	....	−11.7
No. 14867 (V).......	36 160	....	2.0	−2.2	....	0	−4.6	....	0	−2.1	....	0
No. 15433 (V).......	31 520	....	1.5	−13.6	....	−33.4	−8.5	....	+33.4	−6.8	....	+26.7

[a] Average of 5 specimens stored 5 yr. indoors.

[b] Alloys Nos. 14865, 14866, and 14867 (Nos. IVa, Va, and V, respectively) were cast by the Aluminum Company of America. Alloys Nos. 15431, 15432, and 15433 (Nos. IVa, Va, and V, respectively) were cast by the General Electric Co.

[c] Average of 4 specimens; one lost.

Discussion:

Drawing conclusions from the data given in Tables I to III is open to justifiable criticism because the effects of the exposures were relatively slight and the differences from station to station and between the different alloys are believed to be of about the same magnitude as the errors inherent in measuring and recording the results. In all probability, the small differences noted are largely the result of small variations in the properties of the test bars always to be expected in any castings. In fact, it is for this reason that Table II has been prepared.

Table I shows the results reported by each testing laboratory for the specimens tested by it. Since the properties of the "blanks" tested by the two laboratories varied somewhat, differences in the calculated changes in properties of the exposure specimens would be expected even in the absence of corrosion. To avoid this difficulty, the results from the two laboratories were averaged to secure the data of Table II.

It is not believed that the data warrant any conclusion other than that none of the samples has suffered significant corrosion in the period under discussion. Such a result certainly is not surprising in the view of the results previously secured by Subcommittee V.[3]

TABLE II.—INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS NOS. IVa AND Va. AVERAGE RESULTS OF EXPOSURE TO THE ATMOSPHERE FOR 5 YR.

Data are average of 10 specimens without regard to testing laboratory.

Alloy	Original Properties		Sandy Hook, N. J.		New York, N. Y.		Altoona, Pa.	
			Percentage Change		Percentage Change		Percentage Change	
	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength	Elongation in 2 in.	Tensile Strength	Elongation in 2 in.	Tensile Strength	Elongation in 2 in.
No. 14865 (IVa)	27 700	5.9	−2.8	−3.4	−3.1	−3.4	−3.0	+1.7
No. 15431 (IVa)	27 120	5.3	−2.9	−3.8	−3.7	−7.5	−5.6	−11.3
No. 14866 (Va)	33 900	2.8	−3.2	−10.7	−3.5[a]	−25.0[a]	−4.3	−3.6
No. 15432 (Va)	29 120	2.0	−3.3	−5.0	−10.5	−25.0	−5.8	−25.0
No. 14867 (V)	35 760	2.3	−1.4	−4.3	−2.9	−8.7	−1.4	−13.0
No. 15433 (V)	30 830	1.7	−11.5	−23.5	−6.2	+5.9	−3.0	−11.8

[a] Average of 9 specimens.

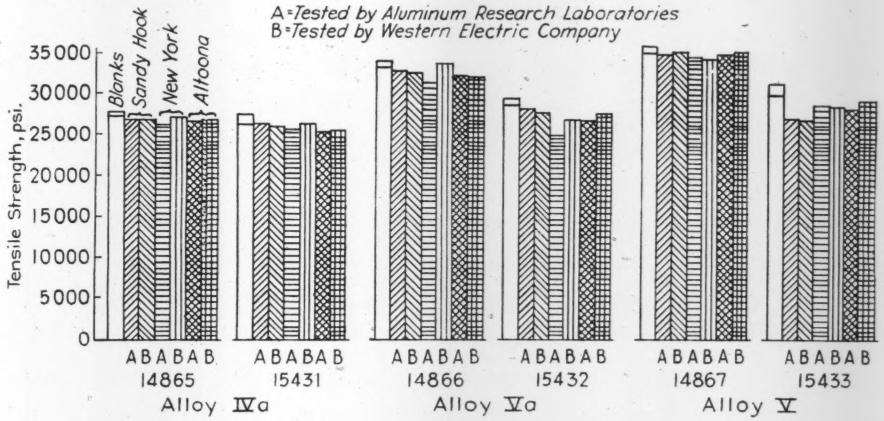

FIG. 1.—Effect of 5-yr. Exposure to the Atmosphere on the Tensile Strength of Aluminum-Alloy Die Castings.

Visual examination of the flat specimens indicated that Sandy Hook was somewhat the least corrosive location and that there was not much difference between the specimens from the New York and Altoona test sites; the latter, however, seemed slightly the more severe. Based on appearance, alloy No. IVa was the most and alloy No. V the least resistant to corrosion at Sandy Hook although the depth of attack measurements (Table III) do not entirely support this observation. There was almost no difference in the appearance of the specimens from the New York and Altoona test sites, largely because the soot and dirt which had collected on the surface masked the true surface of the specimens.

Conclusioms:

The results of the tests and examinations of specimens exposed for 5 yr. at Sandy Hook, New York, and Altoona indicate that alloys Nos. IVa, Va, and V all have a sufficiently high resistance to corrosion that none suffer, in this time, a significant change in mechanical properties. It is therefore not possible to establish any order of merit for the several materials. There is, however, nothing in the present data to suggest that the superior rating given alloy No. Va in the accelerated tests should be modified. Visual examination confirmed the accelerated tests in indicating that the high-purity alloys are more resistant to attack—at least at sea coast locations—than the present commercial grades, although, in industrial locations the soot and dirt deposits mask the small differences in resistance to corrosion which exist at the end of 5 yr. The microscopic measurements recorded in Table III, however, do not entirely support the visual examination, probably because of sampling errors, the type of attack and the limited extent to which the attack has progressed in 5 yr.

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[3] Report of Subcommittee V on Exposure and Corrosion Tests, Appendix II, Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 190 (1935).

TABLE III.—RESULTS OF MICROSCOPIC EXAMINATION OF FLAT SPECIMENS EXPOSED 5 YR.

NOTE.—Measurements made on one transverse section at a magnification of 100×. All specimens were corroded principally by attack of the constituent network.

Alloy	Depth of Attack, in.					
	Sandy Hook, N. J.		New York, N. Y.		Altoona, Pa.	
	Maximum	Average	Maximum	Average	Maximum	Average
No. 14865 (IVa)	0.0067	0.0036	0.0042	0.0034	0.0064	0.0050
No. 15431 (IVa)	0.0042	0.0034	0.0045	0.0036	0.0070	0.0048
No. 14866 (Va)	0.0059	0.0045	0.0048	0.0036	0.0062	0.0048
No. 15432 (Va)	0.0056	0.0039	0.0045	0.0034	0.0070	0.0045
No. 14867 (V)	0.0062	0.0045	0.0064	0.0048	0.0056	0.0042
No. 15433 (V)	0.0056	0.0036	0.0045	0.0039	0.0073	0.0048

Respectfully submitted on behalf of Subcommittee I,

E. E. THUM,
Chairman.

APPENDIX II

TABLE I.—PHYSICAL PROPERTIES AND EXPANSION DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 10-YEAR ATMOSPHERIC EXPOSURE.

Alloy	Producer	Exposure		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼ in. Bars)	Elongation in 2 in., per cent	Brinell Hardness, 500-kg. load, 10-mm. ball, 30 sec.	Expansion, in. per 6 in.
		Type	Location					
No. XIII	C	Outdoors	Key West, Fla.	5 900	0.3	0	61	0.0331
			Sandy Hook, N. J.	17 300	0.4	1	60	0.0065
			New York, N. Y.	16 700	0.2 (I)	1	68 (R)	0.0082
			Altoona, Pa.	17 300	0.4	0	[a]	0.0065
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	[b]	[b]	[b]	[b]	[b]
		Indoors	Coco Solo Islands, C. Z.	12 200	0.3	0	63	0.0238
			New Kensington, Pa.	19 200	0.4	0	64	0.0054
			Cambridge, Mass.	20 100	0.5 (S)	0	67	0.0057
			Hanover, N. Mex.	16 800	0.7	0	78 (R)	0.0026
No. XIII	D	Outdoors	Key West, Fla.	6 600	0.3	[c]	63	0.0257
			Sandy Hook, N. J.	18 100	0.4	[c]	72	0.0076
			New York, N. Y.	19 000	0.3 (I)	[c]	69 (R)	0.0067
			Altoona, Pa.	18 300	0.5	[c]	[a]	0.0066
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	[b]	[b]	[b]	[b]	[b]
		Indoors	Coco Solo Islands, C. Z.	9 800	0.3	[c]	77	0.0225
			New Kensington, Pa.	17 800	0.3	0	71	0.0094
			Cambridge, Mass.	12 900	0.5 (S)	0	72	0.0081
			Hanover, N. Mex.	19 600	0.5	0	82 (R)	0.0038
No. XIII	R	Outdoors	Key West, Fla.	9 200	[b]	0	[b]	[b]
			Sandy Hook, N. J.	14 200	0.3	0	66	0.0077
			New York, N. Y.	10 900	0.3 (I)	1	74 (R)	0.0079
			Altoona, Pa.	17 300	0.3	0	[a]	0.0078
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	[b]	[b]	[b]	[b]	[b]
		Indoors	Coco Solo Islands, C. Z.	11 300	0.2	0	64	0.0199
			New Kensington, Pa.	[c]	0.3	[c]	65	0.0087
			Cambridge, Mass.	13 900	0.5 (S)	0	69	0.0104
			Hanover, N. Mex.	18 700	1.0	0	74 (R)	0.0041
No. XIII	T	Outdoors	Key West, Fla.	8 600	0.2	0	72	0.0197
			Sandy Hook, N. J.	13 200	0.4	0	68	0.0107
			New York, N. Y.	12 900	0.3 (I)	1	78 (R)	0.0089
			Altoona, Pa.	12 600	0.3	[c]	[a]	0.0108
			Rochester, N. Y.	[b]	[b]	[b]	[b]	
			State College, Pa.	[b]	[b]	[b]	[b]	[b]
		Indoors	Coco Solo Islands, C. Z.	8 400	0.2	[c]	79	0.0168
			New Kensington, Pa.	12 000	0.3	0	68	0.0085
			Cambridge, Mass.	10 200	0.5 (S)	0	76	0.0100
			Hanover, N. Mex.	19 200	0.7	1	81 (R)	0.0024
No. XIV	C	Outdoors	Key West, Fla.	10 200	0.2	0	71	0.0671
			Sandy Hook, N. J.	24 600	0.4	0	73	0.0312
			New York, N. Y.	18 200	0.3 (I)	1	82 (R)	0.0337
			Altoona, Pa.	27 300	0.5	0	[a]	0.0338
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	[b]	0.4	[b]	70	0.0257
		Indoors	Coco Solo Islands, C. Z.	12 900	0.2	0	70	0.0771
			New Kensington, Pa.	15 900	0.3	0	72	0.0324
			Cambridge, Mass.	12 100	1.0 (S)	0	75	0.0339
			Hanover, N. Mex.	29 700	0.6	1	83 (R)	0.0225
No. XIV	D	Outdoors	Key West, Fla.	4 900	0.1	0	[c]	0.1812
			Sandy Hook, N. J.	18 700	0.2	0	81	0.0588
			New York, N. Y.	20 700	0.2 (I)	1	87 (R)	0.0529
			Altoona, Pa.	21 700	0.3	0	[a]	0.0480
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	[b]	0.2	[b]	84	0.0469
		Indoors	Coco Solo Islands, C. Z.	[b]	0.2	[b]	[c]	>0.1919
			New Kensington, Pa.	10 900	0.2	0	76	0.0638
			Cambridge, Mass.	9 100	0.3 (S)	0	82	0.0677
			Hanover, N. Mex.	18 600	0.6	0	87 (R)	0.0334

[a] No tests conducted.

[b] Specimens were missing at the exposure location or broken in shipment.

[c] No data reported.

(I)—Izod (impact machine).

(R)—Rockwell hardness, Scale E (⅛-in. ball, 100-kg. load).

(S)—Schopper (impact machine).

TABLE I.—PHYSICAL PROPERTIES AND EXPANSION DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 10-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼ in. Bars)	Elongation in 2 in., per cent	Brinell Hardness, 500-kg. load, 10-mm. ball, 30 sec.	Expansion, in. per 6 in.
		Type	Location					
No. XIV	R	Outdoors	Key West, Fla.	4 900	0.2	0	[c]	>0.2112
			Sandy Hook, N. J.	10 200	0.2	0	70	0.1091
			New York, N. Y.	9 600	0.1 (I)	1	87 (R)	0.0991
			Altoona, Pa.	15 600	0.2	0	[a]	0.0842
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	14 000	0.2	0	68	0.0757
		Indoors	Coco Solo Islands, C. Z.	[b]	[b]	[b]	[b]	>0.2132
			New Kensington, Pa.	4 400	0.2	0	79	0.1168
			Cambridge, Mass.	[b]	0.4 (S)	[b]	72	0.0936
			Hanover, N. Mex.	16 000	0.5	0	88 (R)	0.0533
No. XIV	S	Outdoors	Key West, Fla.	3 800	[b]	0	[b]	[b]
			Sandy Hook, N. J.	25 100	0.3	0	84	0.0376
			New York, N. Y.	27 100	0.3 (I)	1	85 (R)	0.0350
			Altoona, Pa.	25 400	0.3	0	[a]	0.0366
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	25 000	0.3	0	81	0.0306
		Indoors	Coco Solo Islands, C. Z.	[b]	0.1	[b]	72	0.1121
			New Kensington, Pa.	17 400	0.2	0	82	0.0457
			Cambridge, Mass.	17 800	0.4 (S)	0	87	0.0417
			Hanover, N. Mex.	25 300	0.6	0	89 (R)	0.0251
No. XIV	T	Outdoors	Key West, Fla.	8 700	0.2	0	[c]	0.1101
			Sandy Hook, N. J.	20 900	0.3	0	68	0.0411
			New York, N. Y.	18 100	0.3 (I)	1	82 (R)	0.0438
			Altoona, Pa.	20 500	0.3	0	[a]	0.0403
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	20 500	0.3	1	66	0.0378
		Indoors	Coco Solo Islands, C. Z.	[b]	0.2	[b]	75	0.1105
			New Kensington, Pa.	13 000	0.2	0	[c]	0.0419
			Cambridge, Mass.	11 800	0.4 (S)	0	72	0.0411
			Hanover, N. Mex.	16 100	1.7	0	81 (R)	0.0275
No. XV	C	Outdoors	Key West, Fla.	29 800	0.4	0	87	0.0227
			Sandy Hook, N. J.	47 200	0.8	1	98	0.0091
			New York, N. Y.	46 100	1.3 (I)	1	83 (R)	0.0084
			Altoona, Pa.	49 200	1.3	1	[a]	0.0087
			Rochester, N. Y.	[b]	1.3	[b]	95 (R)	0.0074
			State College, Pa.	51 700	1.6	1	93	0.0036
		Indoors	Coco Solo Islands, C. Z.	43 300	0.7	1	86	0.0147
			New Kensington, Pa.	49 500	1.0	1	93	0.0119
			Cambridge, Mass.	50 300	1.4 (S)	1	95	0.0078
			Hanover, N. Mex.	50 400	1.4	1	96 (R)	0.0071
No. XV	D	Outdoors	Key West, Fla.	25 100	0.3	1	87	0.0356
			Sandy Hook, N. J.	41 600	0.5	1	100	0.0138
			New York, N. Y.	39 900	0.4 (I)	1	87 (R)	0.0138
			Altoona, Pa.	41 500	0.8	0	[a]	0.0126
			Rochester, N. Y.	[b]	0.7	[b]	97 (R)	0.0105
			State College, Pa.	44 500	0.9	1	103	0.0086
		Indoors	Coco Solo Islands, C. Z.	22 700	0.3	1	85	0.0317
			New Kensington, Pa.	39 200	0.5	0	98	0.0176
			Cambridge, Mass.	37 000	0.5 (S)	0	106	0.0142
			Hanover, N. Mex.	42 400	0.8	0	96 (R)	0.0100
No. XV	R	Outdoors	Key West, Fla.	33 500	0.4	0	94	0.0233
			Sandy Hook, N. J.	54 100	1.5	1	107	0.0058
			New York, N. Y.	51 200	1.9 (I)	1	90 (R)	0.0065
			Altoona, Pa.	51 200	1.4	1	[a]	0.0068
			Rochester, N. Y.	[b]	1.8	[b]	99 (R)	0.0036
			State College, Pa.	54 500	1.7	2	99	0.0029
		Indoors	Coco Solo Islands, C. Z.	37 600	0.6	0	95	0.0210
			New Kensington, Pa.	45 500	0.9	1	97	0.0125
			Cambridge, Mass.	47 700	1.7 (S)	0	112	0.0079
			Hanover, N. Mex.	49 900	1.7	1	104 (R)	0.0063

[a] No tests conducted.

[b] Specimens were missing at the exposure location or broken in shipment.

[c] No data reported.

(I)—Izod (impact machine).

(R)—Rockwell hardness, Scale E (⅛-in. ball, 100-kg. load).

(S)—Schopper (impact machine).

TABLE I.—PHYSICAL PROPERTIES AND EXPANSION DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 10-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼ in. Bars)	Elongation in 2 in., per cent	Brinell Hardness, 500-kg. load, 10-mm. ball, 30 sec.	Expansion, in. per 6 in.
		Type	Location					
No. XV	S	Outdoors	Key West, Fla.	23 000	0.4	0	87	0.0317
			Sandy Hook, N. J.	40 000	0.6	1	98	0.0103
			New York, N. Y.	38 000	0.6 (I)	1	84 (R)	0.0089
			Altoona, Pa.	43 800	0.8	0	[a]	0.0097
			Rochester, N. Y.	[b]	0.8	[b]	96 (R)	0.0074
			State College, Pa.	45 000	1.0	1	101	0.0054
		Indoors	Coco Solo Islands, C. Z.	29 100	0.5	0	91	0.0259
			New Kensington, Pa.	41 200	0.7	0	98	0.0139
			Cambridge, Mass.	39 500	0.8 (S)	0	109	0.0094
			Hanover, N. Mex.	45 200	1.2	0	97 (R)	0.0065
No. XV	Z	Outdoors	Key West, Fla.	19 500	0.3	0	84	0.0498
			Sandy Hook, N. J.	34 600	0.5	0	94	0.0164
			New York, N. Y.	33 800	0.5 (I)	1	83 (R)	0.0173
			Altoona, Pa.	37 600	0.5	0	[a]	0.0156
			Rochester, N. Y.	[b]	0.6	[b]	95 (R)	0.0107
			State College, Pa.	39 000	0.6	1	95	0.0105
		Indoors	Coco Solo Islands, C. Z.	13 000	0.3	0	83	0.0568
			New Kensington, Pa.	33 200	0.3	1	92	0.0200
			Cambridge, Mass.	29 400	0.4 (S)	0	95	0.0154
			Hanover, N. Mex.	37 300	0.6	0	96 (R)	0.0099
No. XVI	C	Outdoors	Key West, Fla.	30 900	0.4	1	80	0.0313
			Sandy Hook, N. J.	48 900	0.9	1	97	0.0117
			New York, N. Y.	46 300	1.2 (I)	1	84 (R)	0.0116
			Altoona, Pa.	46 000	1.3	0	[a]	0.0110
			Rochester, N. Y.	[b]	1.3	[b]	95 (R)	0.0088
			State College, Pa.	50 300	1.1	1	92	0.0079
		Indoors	Coco Solo Islands, C. Z.	41 600	0.6	1	82	0.0177
			New Kensington, Pa.	44 600	1.0	1	83	0.0137
			Cambridge, Mass.	47 200	1.1 (S)	1	97	0.0114
			Hanover, N. Mex.	49 100	1.3	1	95 (R)	0.0112
No. XVI	D	Outdoors	Key West, Fla.	28 200	0.4	0	92	0.0288
			Sandy Hook, N. J.	42 100	0.6	0	100	0.0130
			New York, N. Y.	41 900	0.7 (I)	1	87 (R)	0.0128
			Altoona, Pa.	46 400	1.0	0	[a]	0.0125
			Rochester, N. Y.	[b]	0.8	[b]	97 (R)	0.0095
			State College, Pa.	49 500	0.8	2	104	0.0067
		Indoors	Coco Solo Islands, C. Z.	29 900	0.4	1	88	0.0285
			New Kensington, Pa.	43 900	0.7	0	98	0.0165
			Cambridge, Mass.	43 700	0.9 (S)	0	107	0.0124
			Hanover, N. Mex.	48 200	1.4	1	97 (R)	0.0106
No. XVI	S	Outdoors	Key West, Fla.	20 600	0.4	0	89	0.0343
			Sandy Hook, N. J.	38 300	0.6	0	102	0.0117
			New York, N. Y.	36 100	0.5 (I)	1	85 (R)	0.0120
			Altoona, Pa.	38 300	0.8	0	[a]	0.0086
			Rochester, N. Y.	[b]	0.9	[b]	95 (R)	0.0075
			State College, Pa.	43 200	0.8	1	99	0.0064
		Indoors	Coco Solo Islands, C. Z.	14 000	0.3	0	93	0.0352
			New Kensington, Pa.	34 800	0.6	0	98	0.0175
			Cambridge, Mass.	38 300	0.9 (S)	0	104	0.0096
			Hanover, N. Mex.	42 700	2.7	1	95 (R)	0.0073
No. XVI	Z	Outdoors	Key West, Fla.	24 100	0.5	0	81	0.0211
			Sandy Hook, N. J.	40 200	1.1	1	86	0.0081
			New York, N. Y.	40 300	1.3 (I)	1	82 (R)	0.0087
			Altoona, Pa.	41 400	1.6	0	[a]	0.0044
			Rochester, N. Y.	[b]	1.9	[b]	93 (R)	0.0052
			State College, Pa.	46 300	1.8	1	92	0.0040
		Indoors	Coco Solo Islands, C. Z.	37 200	0.7	0	88	0.0146
			New Kensington, Pa.	43 600	1.0	1	86	0.0105
			Cambridge, Mass.	43 800	1.7 (S)	1	93	0.0072
			Hanover, N. Mex.	43 300	1.2	1	94 (R)	0.0047

[a] No tests conducted.

[b] Specimens were missing at the exposure location or broken in shipment.

[c] No data reported.

(I)—Izod (impact machine).

(R)—Rockwell hardness, Scale E (⅛-in. ball, 100-kg. load).

(S)—Schopper (impact machine).

TABLE I.—PHYSICAL PROPERTIES AND EXPANSION DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 10-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼ in. Bars)	Elongation in 2 in., per cent	Brinell Hardness, 500-kg. load, 10-mm. ball, 30 sec.	Expansion, in. per 6 in.
		Type	Location					
No. XVII	D	Outdoors	Key West, Fla.	22 200	0.3	0	94	0.0432
			Sandy Hook, N. J.	33 500	0.4	0	97	0.0092
			New York, N. Y.	25 200	0.5 (I)	1	84 (R)	0.0101
			Altoona, Pa.	30 500	0.7	0	[a]	0.0072
			Rochester, N. Y.	[b]	0.5	[b]	97 (R)	0.0068
			State College, Pa.	39 800	0.7	1	104	0.0050
		Indoors	Coco Solo Islands, C. Z.	25 000	0.4	1	91	0.0202
			New Kensington, Pa.	34 600	0.6	0	100	0.0111
			Cambridge, Mass.	31 700	0.5 (S)	0	106	0.0078
			Hanover, N. Mex.	34 200	0.8	0	97 (R)	0.0052
No. XVII	S	Outdoors	Key West, Fla.	18 200	0.4	0	96	0.0360
			Sandy Hook, N. J.	33 500	0.5	0	96	0.0109
			New York, N. Y.	33 400	0.5 (I)	1	89 (R)	0.0123
			Altoona, Pa.	37 300	0.6	0	[a]	0.0080
			Rochester, N. Y.	[b]	0.6	[b]	96 (R)	0.0059
			State College, Pa.	39 400	0.7	1	97	0.0049
		Indoors	Coco Solo Islands, C. Z.	25 700	0.4	0	93	0.0293
			New Kensington, Pa.	35 200	0.5	0	99	0.0122
			Cambridge, Mass.	35 900	0.5 (S)	0	110	0.0084
			Hanover, N. Mex.	37 800	1.0	0	96 (R)	0.0049
No. XVII	T	Outdoors	Key West, Fla.	18 500	0.3	0	85	0.0441
			Sandy Hook, N. J.	30 000	0.3	0	101	0.0121
			New York, N. Y.	26 500	0.2 (I)	1	85 (R)	0.0125
			Altoona, Pa.	32 600	0.5	0	[a]	0.0081
			Rochester, N. Y.	[b]	0.6	[b]	96 (R)	0.0064
			State College, Pa.	35 400	0.8	0	98	0.0052
		Indoors	Coco Solo Islands, C. Z.	27 300	0.4	0	92	0.0222
			New Kensington, Pa.	34 900	0.5	1	94	0.0115
			Cambridge, Mass.	36 100	0.6 (S)	0	108	0.0075
			Hanover, N. Mex.	34 800	1.0	0	95 (R)	0.0051
No. XVIII	C	Outdoors	Key West, Fla.	13 100	0.3	1	48	0.0356
			Sandy Hook, N. J.	20 600	0.4	1	45	0.0160
			New York, N. Y.	16 600	0.3 (I)	1	59 (R)	0.0123
			Altoona, Pa.	18 300	0.4	0	[a]	0.0155
			Rochester, N. Y.	[b]	0.5	[b]	62 (R)	0.0154
			State College, Pa.	19 200	0.4	1	50	0.0101
		Indoors	Coco Solo Islands, C. Z.	11 400	0.3	0	48	0.0276
			New Kensington, Pa.	17 500	0.3	1	51	0.0131
			Cambridge, Mass.	16 500	0.6 (S)	0	54	0.0090
			Hanover, N. Mex.	19 200	1.3	0	64 (R)	0.0133
No. XVIII	S	Outdoors	Key West, Fla.	10 600	0.3	1	[c]	0.0541
			Sandy Hook, N. J.	11 500	0.4	0	58	0.0269
			New York, N. Y.	9 400	0.5 (I)	1	62 (R)	0.0259
			Altoona, Pa.	8 100	0.4	0	[a]	0.0207
			Rochester, N. Y.	[b]	0.6	[b]	70 (R)	0.0173
			State College, Pa.	9 900	0.4	0	55	0.0127
		Indoors	Coco Solo Islands, C. Z.	[c]	0.3	[c]	62	0.0536
			New Kensington, Pa.	12 400	0.4	0	58	0.0242
			Cambridge, Mass.	10 800	0.6 (S)	0	51	0.0236
			Hanover, N. Mex.	11 100	0.5	0	71 (R)	0.0218
No. XVIII	T	Outdoors	Key West, Fla.	8 500	0.2	0	38	0.0428
			Sandy Hook, N. J.	14 900	0.3	0	55	0.0217
			New York, N. Y.	14 600	0.2 (I)	1	65 (R)	0.0201
			Altoona, Pa.	14 100	0.4	0	[a]	0.0213
			Rochester, N. Y.	[b]	0.4	[b]	70 (R)	0.0186
			State College, Pa.	17 900	0.3	[c]	52	0.0180
		Indoors	Coco Solo Islands, C. Z.	[b]	0.2	[b]	58	0.0407
			New Kensington, Pa.	12 400	0.3	1	61	0.0182
			Cambridge, Mass.	14 900	0.5 (S)	0	61	0.0146
			Hanover, N. Mex.	15 100	0.4	0	70 (R)	0.0094

[a] No tests conducted.

[b] Specimens were missing at the exposure location or broken in shipment.

[c] No data reported.

(I)—Izod (impact machine).

(R)—Rockwell hardness, Scale E (⅛-in. ball, 100-kg. load).

(S)—Schopper (impact machine).

TABLE I.—PHYSICAL PROPERTIES AND EXPANSION DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 10-YEAR ATMOSPHERIC EXPOSURE.—*Concluded.*

Alloy	Producer	Exposure		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼ in. Bars)	Elongation in 2 in., per cent	Brinell Hardness, 500-kg. load, 10-mm. ball, 30 sec.	Expansion, in. per 6 in.
		Type	Location					
No. XIX	D	Outdoors	Key West, Fla.	23 500	0.4	0	77	0.0221
			Sandy Hook, N. J.	34 900	0.9	1	82	0.0052
			New York, N. Y.	32 200	1.4 (I)	1	78 (R)	0.0058
			Altoona, Pa.	35 800	1.7	1	[a]	0.0055
			Rochester, N. Y.	[b]	1.4	[b]	87 (R)	0.0039
			State College, Pa.	39 500	1.5	1	82	0.0021
		Indoors	Coco Solo Islands, C. Z.	24 200	0.5	1	78	0.0191
			New Kensington, Pa.	37 800	0.7	1	84	0.0063
			Cambridge, Mass.	37 800	1.3 (S)	1	85	0.0030
			Hanover, N. Mex.	39 100	1.2	2	89 (R)	0.0017
No. XIX	R	Outdoors	Key West, Fla.	13 300	0.4	0	82	0.0251
			Sandy Hook, N. J.	35 000	1.6	0	86	0.0025
			New York, N. Y.	33 600	1.9 (I)	1	84 (R)	0.0022
			Altoona, Pa.	36 000	2.3	1	[a]	0.0028
			Rochester, N. Y.	[b]	3.1	[b]	89 (R)	0.0009
			State College, Pa.	39 200	2.4	2	83	−0.0001
		Indoors	Coco Solo Islands, C. Z.	18 400	0.4	0	86	0.0257
			New Kensington, Pa.	34 500	0.9	1	89	0.0036
			Cambridge, Mass.	35 000	1.8 (S)	0	86	0.0012
			Hanover, N. Mex.	38 000	2.5	1	91 (R)	−0.0006
No. XIX	Z	Outdoors	Key West, Fla.	23 300	0.7	1	71	0.0116
			Sandy Hook, N. J.	33 500	2.1	1	71	0.0024
			New York, N. Y.	30 500	2.8	1	76 (R)	0.0030
			Altoona, Pa.	33 300	4.0	1	[a]	0.0073
			Rochester, N. Y.	[b]	4.4	[b]	83 (R)	0.0029
			State College, Pa.	36 500	3.5	3	74	0.0012
		Indoors	Coco Solo Islands, C. Z.	33 400	1.6	1	73	0.0050
			New Kensington, Pa.	35 900	3.8	2	71	0.0014
			Cambridge, Mass.	36 400	6.5 (S)	3	74	0.0009
			Hanover, N. Mex.	35 700	3.3	1	83 (R)	0.0006
No. XX	C	Outdoors	Key West, Fla.	11 500	[b]	0	[b]	[b]
			Sandy Hook, N. J.	[c]	0.4	[c]	62	0.0408
			New York, N. Y.	27 600	0.3 (I)	1	69 (R)	0.0474
			Altoona, Pa.	28 400	0.5	0	[a]	0.0294
			Rochester, N. Y.	[b]	0.4	[b]	84 (R)	0.0433
			State College, Pa.	34 200	0.3	[c]	63	0.0266
		Indoors	Coco Solo Islands, C. Z.	[b]	[b]	[b]	[b]	[b]
			New Kensington, Pa.	23 800	0.3	0	61	0.0643
			Cambridge, Mass.	24 000	0.9 (S)	0	66	0.0480
			Hanover, N. Mex.	30 200	0.4	0	84 (R)	0.0390
No. XX	D	Outdoors	Key West, Fla.	4 700	[b]	0	[b]	[c]
			Sandy Hook, N. J.	26 300	0.3	1	85	0.0737
			New York, N. Y.	23 400	0.3 (I)	1	88 (R)	0.0716
			Altoona, Pa.	27 700	0.4	0	[a]	0.0622
			Rochester, N. Y.	[b]	0.4	[b]	88 (R)	0.0579
			State College, Pa.	27 900	0.3	0	84	0.0575
		Indoors	Coco Solo Islands, C. Z.	[b]	[b]	[b]	[b]	[b]
			New Kensington, Pa.	8 600	0.2	0	83	0.1431
			Cambridge, Mass.	16 100	0.4 (S)	0	85	0.0656
			Hanover, N. Mex.	19 800	0.4	0	90 (R)	0.0440
No. XXII	C	Outdoors	Key West, Fla.	11 300	0.3	0	68	0.0567
			Sandy Hook, N. J.	13 900	0.3	1	50	0.0261
			New York, N. Y.	14 400	0.1 (I)	1	63 (R)	0.0275
			Altoona, Pa.	15 300	0.4	0	[a]	0.0403
			Rochester, N. Y.	[b]	[b]	[b]	[b]	[b]
			State College, Pa.	[b]	[b]	[b]	[b]	[b]
		Indoors	Coco Solo Islands, C. Z.	8 300	0.2	[c]	55	0.0395
			New Kensington, Pa.	12 800	0.3	0	52	0.0209
			Cambridge, Mass.	12 900	0.8 (S)	0	63	0.0206
			Hanover, N. Mex.	13 300	0.4	0	69 (R)	0.0198

[a] No tests conducted.

[b] Specimens were missing at the exposure location or broken in shipment.

[c] No data reported.

(I)—Izod (impact machine).

(R)—Rockwell hardness, Scale E (⅛-in. ball, 100-kg. load).

(S)—Schopper (impact machine).

TABLE II.—EXPANSION DATA ON ZINC-BASE ALLOY NO. XXI AFTER 10-YEAR ATMOSPHERIC EXPOSURE.[a]

Alloy	Producer	Exposure		Expansion, in. per 6 in.
		Type	Location	
No. XXI	D	Outdoors	Key West, Fla.	0.0152
			Sandy Hook, N. J.	0.0069
			New York, N. Y.	0.0067
			Altoona, Pa.	0.0085
			Rochester, N. Y.	0.0061
			State College, Pa.	0.0064
		Indoors	Coco Solo Islands, C. Z.	[b]
			New Kensington, Pa.	0.0108
			Cambridge, Mass.	0.0089
			Hanover, N. Mex.	0.0085
No. XXI	R	Outdoors	Key West, Fla.	0.0137
			Sandy Hook, N. J.	0.0044
			New York, N. Y.	0.0049
			Altoona, Pa.	0.0040
			Rochester, N. Y.	0.0032
			State College, Pa.	0.0013
		Indoors	Coco Solo Islands, C. Z.	0.0113
			New Kensington, Pa.	0.0098
			Cambridge, Mass.	0.0072
			Hanover, N. Mex.	0.0062
No. XXI	S	Outdoors	Key West, Fla.	0.0099
			Sandy Hook, N. J.	0.0028
			New York, N. Y.	0.0015
			Altoona, Pa.	0.0101
			Rochester, N. Y.	0.0018
			State College, Pa.	0.0018
		Indoors	Coco Solo Islands, C. Z.	0.0076
			New Kensington, Pa.	0.0056
			Cambridge, Mass.	0.0038
			Hanover, N. Mex.	0.0035
No. XXI	T	Outdoors	Key West, Fla.	0.0153
			Sandy Hook, N. J.	0.0079
			New York, N. Y.	0.0074
			Altoona, Pa.	0.0087
			Rochester, N. Y.	0.0061
			State College, Pa.	0.0062
		Indoors	Coco Solo Islands, C. Z.	0.0140
			New Kensington, Pa.	0.0105
			Cambridge, Mass.	0.0099
			Hanover, N. Mex.	0.0088
No. XXI	Z	Outdoors	Key West, Fla.	0.0145
			Sandy Hook, N. J.	0.0031
			New York, N. Y.	0.0039
			Altoona, Pa.	0.0081
			Rochester, N. Y.	0.0029
			State College, Pa.	0.0027
		Indoors	Coco Solo Islands, C. Z.	0.0094
			New Kensington, Pa.	0.0086
			Cambridge, Mass.	0.0056
			Hanover, N. Mex.	0.0066

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[a] Specimens of alloy No. XXI are being re-exposed for an indefinite period and accordingly the tests for physical properties were not made at the end of the 10-yr. exposure period.

[b] Specimens were missing at the exposure location or broken in shipment.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 on Light Metals and Alloys has held two meetings during the past year: the first in Atlantic City, N. J., on June 29, 1939, and the more recent in Detroit, Mich. on March 7, 1940.

Membership changes since the 1939 annual meeting have included the appointment of three new members and the reappointment of three members previously dropped because of change in employment. One resignation has been received. In addition, the classification of three members has been corrected. The membership of the committee now totals 47, of whom 21 are classified as producers, 18 as consumers, and 8 as general interest members.

Several new subcommittee appointments have been made, including representatives of the American Foundrymen's Association to replace their former representative, Sam Tour. Also, J. D. Edwards, Aluminum Research Laboratories, was appointed chairman of Subcommittee VI to replace T. S. Fuller who resigned.

Subsequent to the 1939 annual meeting, Committee B-7 submitted to the Society through Committee E-10 on Standards new Tentative Specifications for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 39 T) and a revision of the Tentative Method of Test for Dielectric Strength of Anodized Aluminum (B 110 – 38 T). The new tentative specifications were prepared in response to a request by the Boiler Code Committee of the American Society of Mechanical Engineers, which committee also cooperated with Subcommittee III in the preparation of the specifications. The revisions recommended in Tentative Method B 110 – 38 T were prompted by comments received by Subcommittee VI as a result of the initial distribution of this method. These recommendations were accepted[1] by Committee E-10 on August 24, 1939, and the new and revised tentative standards appear in the 1939 Book of A.S.T.M. Standards.[2]

The following papers, prepared under the direction of Subcommittee VI are being sponsored by Committee B-7 for presentation at this annual meeting of the Society:[3]

- Anodic Coatings Seen Through the Microscope, by F. Keller, Aluminum Company of America,
- Thickness of Anodic Coatings on Aluminum, by Junius D. Edwards, Aluminum Company of America,
- The Abrasion Resistance of Anodically Oxidized Coatings on Aluminum, by H. G. Arlt, Bell Telephone Laboratories, Inc., and
- Electrical Breakdown of Anodically Oxidized Coatings on Aluminum: A Means of Checking Thickness of Anodized Finishes, by K. G. Compton and A. Mendizza, Bell Telephone Laboratories, Inc.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-7 reported the following results of the letter ballot vote of a total of 27 ballots returned from a committee membership of 44: B 126 – 39 T, affirmative 17, negative 0, ballots marked "not voting" 10; B 110 - 38 T, affirmative 19, negative 0, ballots marked "not voting" 8.

[2] 1939 Book of A.S.T.M. Standards, Part I, pp. 1031, 1040.

[3] See pp. 948, 959, 967, and 978, respectively.

I. NEW TENTATIVE STANDARDS

The committee is submitting for publication as tentative the following two new methods:[4]

Tentative Methods of:

Test for Weight of Coating on Anodically Coated Aluminum, and

Test for Sealing of Anodically Coated Aluminum.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following eleven tentative specifications be revised and continued as tentative. Four of the specifications are appended in their revised form,[5] while the revisions of the remaining seven are indicated below. These revisions are recommended in order to bring the specifications in line with present commercial practices:

Tentative Specifications for:

Magnesium-Base Alloy Ingot for Remelting (B 93 – 39 T),[6]

Magnesium-Base Alloy Sand Castings (B 80 – 38 T),[6]

Magnesium-Base Alloy Forgings (B 91 – 38 T),[6]

Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 38 T).[6]

Magnesium-Base Alloy Sheet (B 90 – 38 T),[6] revised as follows:

Section 2.—Change from its present form: namely,

> 2. The alloys shall be made from virgin metal or from purified scrap metal of known composition.

to read as follows:

> 2. The materials used shall be such as to produce products that will conform to the requirements as to chemical composition and physical properties prescribed in these specifications.

Table I.—Add the following two notes under the title of this table:

> NOTE 1.—Analysis shall regularly be made only for the elements specifically mentioned in this table. If, however, the presence of other elements is suspected, or indicated in the course of routine analysis, further analysis shall be made to determine that the total of these other elements is not in excess of the limits specified in the last column of the table.
>
> NOTE 2.—The following applies to all specified limits in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained from analysis should be rounded off to the nearest unit in the last right-hand place of figures used in expressing the specified limit.

In place of the present footnote *a* in this table, add the requirements for nickel to the table, as follows:

Alloy	Nickel, max., per cent
No. 6	0.03
No. 7	0.03
No. 11	0.03

Aluminum Sheet and Plate (B 25 – 38 T).[6]

Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum - Copper - Magnesium - Manganese) (B 78 – 39 T).[6]

Aluminum-Manganese Alloy Sheet and Plate (B 79 – 38 T).[6]

Aluminum-Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 – 39 T).[6]

Aluminum - Magnesium - Chromium Alloy Sheet and Plate (B 109 – 39 T).[6]

Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 39 T).[6]

In the last ten specifications listed above the following change is recommended:

Methods of Testing.—Add the following as a new section on methods of testing:

> The tension tests shall be made in accordance with the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.

Fig. 1.—Change the note regarding the radius at the end of the reduced section from "radius not less than $\frac{1}{8}$ in." to read "radius not less than $\frac{3}{8}$ in."

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[4] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, pp. 339 and 338.

[5] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, pp. 341, 343, 355, and 347, respectively.

[6] 1939 Book of A.S.T.M. Standards, Part I, pp. 1042 to 1058, incl. and 1013 to 1034, incl.

III. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The committee also recommends, in view of a review that still needs to be made, that the following four specifications be continued as tentative without revision:

Tentative Specifications for:

Aluminum-Base Alloy Sand Castings (B 26 – 37 T),

Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 – 37 T),

Aluminum-Base Alloy Permanent Mold Castings (B 108 – 38 T), and

Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112 – 38 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 47 members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (D. L. Colwell, chairman).—During the past year this subcommittee has reviewed the various specifications under its jurisdiction. In the absence of any demands for revisions, the subcommittee recommends that these specifications be continued as tentative. This subcommittee has also considered the advisability of preparing specifications for various types of aluminum hardeners, but no definite action has as yet been taken.

Subcommittee II on Aluminum Alloy Castings (C. H. Greenall, chairman).—A review of the various specifications under the jurisdiction of this subcommittee has shown no need for revisions at this time. It has therefore been recommended that these specifications be continued as tentative until the necessary data can be collected to justify changes. To facilitate the collection of such data, the original subgroup appointed to study the effect of minor elements on the characteristics of several specific alloys has been replaced by two subgroups with broader scopes. One subgroup will study the sand casting alloys and the other the permanent mold alloys. It is hoped that this study, which constitutes one of the major problems of this subcommittee for the next year, will also clarify certain composition differences which now exist between Federal and A.S.T.M. specifications.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Weight of Coating on Anodically Coated Aluminum	29	0	8
Test for Sealing of Anodically Coated Aluminum	29	0	8
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Magnesium-Base Alloy Ingot for Remelting (B 93 – 39 T)	27	0	10
Spec. for Magnesium-Base Alloy Sand Castings (B 80 – 38 T)	32	0	4
Spec. for Magnesium-Base Alloy Forgings (B 91 – 38 T)	29	0	7
Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 38 T)	29	0	7
Spec. for Magnesium-Base Alloy Sheet (B 90 – 38 T)	29	0	7
Spec. for Aluminum Sheet and Plate (B 25 – 38 T)	30	0	6
Spec. for Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum - Copper - Magnesium-Manganese) (B 78 – 39 T)	30	0	6
Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 – 38 T)	30	0	6
Spec. for Aluminum-Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum - Copper - Magnesium-Manganese (B 89 – 39 T)	30	0	6
Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 39 T)	29	0	7
Spec. for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 39 T)	29	0	7

During the year the subcommittee has also considered a request from a producer for specifications for a new patented aluminum casting alloy. A study of this request failed to show the necessary consumer demand, and the producer was advised that such a demand would have to be established before any action would be taken by the subcommittee.

Subcommittee III on Aluminum Alloy Wrought Shapes (P. V. Faragher, chairman).—The specifications under the jurisdiction of this subcommittee have been reviewed and no need for revisions at this time was recorded. It has therefore been recommended that such specifications be continued as tentative. The subcommittee has under advisement the possibility of consolidating certain of its specifications and plans to review this matter during the coming year.

Subcommittee IV on Magnesium and Magnesium Alloys (J. A. Gann, chairman.)—A review of the various specifications under the jurisdiction of this subcommittee has indicated that certain changes and additions are necessary in several cases to bring the specifications into closer agreement with present commercial practices. Revisions, as mentioned earlier in this report, are therefore being recommended in the Tentative Specifications for Magnesium-Base Alloy Sand Castings (B 80 - 38 T), Sheet (B 90 - 38 T), Forgings (B 91 - 38 T), Ingot for Remelting (B 93 - 39 T), and Bars, Rods, and Shapes (B 107 - 38 T).

Among the changes included in these revisions are the addition of several new casting and wrought alloys, certain of which have better resistance to corrosion and better working characteristics than alloys covered by the present specifications. Two wrought alloys have been deleted since they are no longer used commercially. In addition, the requirements with respect to the material used to produce these alloys and the chemical analysis have been clarified and made to conform to the requirements of other specifications under the jurisdiction of Committee B-7.

The subcommittee is planning, as one of its problems for the coming year, the preparation of specifications for protective coatings for magnesium-base alloy sand castings, forgings, sheet, and rod.

Subcommittee V on Testing Light Alloys (R. L. Templin, chairman).—The tests on the various light alloys supplied by this subcommittee in connection with the work being sponsored by the Section on Effect of Speed of Testing of Committee E-1 on Methods of Testing have been completed. A detailed report of these tests, which will conclude this coöperative program, will be presented at this annual meeting of the Society in the form of a paper by Paul G. Jones and H. F. Moore, entitled "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals."[7]

The subcommittee has two problems for consideration during the coming year: (*1*) a review of the methods of testing included in the various specifications for light metals and alloys is planned in an effort to standardize the procedures; and (*2*) the possibility of using the "divider method" as a means of determining the yield strength of these materials will also be investigated.

Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys (J. D. Edwards, chairman).—This subcommittee and particularly the Subgroup on Methods of Testing Anodic Coatings have continued their investigations of methods for determining the various characteristics of such coatings. This work has led to the development of two new methods which are being recommended for publication as tentative: namely, Test for Weight of Coating on Anodically Coated Aluminum, and Test for Sealing of Anodically Coated Aluminum.[4] The activity of this subgroup has also been responsible for the preparation of the four papers on the subject of testing of oxide coatings which Committee B-7 is sponsoring at this annual meeting of the Society, as mentioned earlier in this report.

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[7] See p. 610.

Future work of this subcommittee, some of which is already in progress, includes: the development of methods for determining abrasion resistance and thickness of anodic coatings; the application of the salt spray test for determining the quality of coatings; and methods of testing scratch resistance, smudge resistance, reflectivity, and paint adhesion.

Subcommittee VII on General Information and Publicity (E. H. Dix, Jr., chairman).—This subcommittee is still considering the question of revising the Report on Service Characteristics of the Light Metals and Their Alloys, published in 1934.[8] Although a previous majority opinion returned by the subcommittee did not favor revision or continuation of this report, a more general survey, including the members of Committee B-7 and other interested members of the Society, is being made. The subcommittee hopes to have a final report on this question available shortly.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 47 members; 37 members returned their ballots, of whom 36 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SAM TOUR,
Chairman.

H. J. ROWE,
Secretary.

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[8] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 277 (1934); also issued as separate publication.

REPORT OF COMMITTEE C-1
ON
CEMENT

The preparation of tentative specifications embracing five types of portland cement constituted the major activity of Committee C-1 in the past year. Several years ago the committee authorized the appointment of a special committee, composed of other than the producing interests, to report on the number of types of portland cement and their nature, which it believed would fulfill the demands of the concrete industry. At the fall meeting of Committee C-1 the special committee presented an interesting report, including Proposed Specifications for Portland Cement.[1] After an extended discussion this was ordered distributed to the committee to be a matter of consideration and action at the spring meeting. At this latter time it was decided, subject to letter ballot of the committee, to present the specifications to the Society for publication as tentative,[2] to become effective September 2, 1940. These specifications are ultimately to replace the present Standard Specifications for Portland Cement (C 9 – 38) and for High-Early-Strength Portland Cement (C 74 – 39).

Mr. D. Wolochow, vice-chairman of Committee C-1, proposed last year that a meeting of the committee devoted to a Symposium on Cement Specifications and Additions to Portland Cement would be of great help to the membership. The suggestion appealed to all, and Mr. Wolochow was instructed to proceed in carrying out his idea. As a result of his activity and the assistance of F. H. Jackson and H. J. Gilkey, a very valuable group of 17 papers was prepared. The presentation and discussion of these occupied one day of the spring meeting of the committee. Since it had been possible to distribute the papers previous to the meeting, there was much valuable discussion. It is not the intention to publish the papers comprising the symposium, but a limited number of mimeographed copies have been made available which may be secured from Mr. Wolochow at a nominal cost.

The Working Committees on Methods of Chemical Analysis (W. C. Hanna, chairman), Plastic Mortar Tests (O. L. Moore, chairman), and Volume Change and Soundness (H. F. Gonnerman, chairman), advanced their work sufficiently to justify the publication of some of the results. The reports of these committees are appended hereto.

The Working Committee on Volume Change and Soundness has also revised the method for determining soundness of cements by the autoclave. This is being presented to the Society for publication as tentative under the title "Tentative Method of Test for Autoclave Expansion of Portland Cement."[3]

Committee C-1 has approved the following four recommendations of the Working Committee on Methods of Chemical Analysis: (*1*) Adopt as standard Sections 1 to 9, inclusive, of the

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[1] "How Many Specifications for Cement?" ASTM BULLETIN, No. 102, January, 1940, p. 39.

[2] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 167.

[3] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 176.

Tentative Methods of Chemical Analysis of Portland Cement (C 114 – 39 T),[4] with two minor changes, the addition of a note to Section 2 and a revision of the first eight sentences of Section 5 to provide for the removal of silicon dioxide.[5]

These sections cover the determination of sodium oxide, potassium oxide, water-soluble alkali, phosphorus pentoxide, and manganic oxide. (*2*) Revise Tentative Methods C 114 – 39 T by the addition of a quick method (ammonium chloride method) for the determination of silicon dioxide, calcium oxide, and magnesium oxide.[6] (*3*) Adopt as standard an alternate procedure for determining phosphorus pentoxide in portland cement.[7] (*4*) Adopt as standard a procedure for determining chloroform-soluble organic substances in portland cement.[7]

As a result of the study thus far made of the recently completed tests reported by the Working Committee on Plastic Mortar Tests, and pending the results of some further work, it is recommended that the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 37 T) be retained as tentative without change at this time.

The Subcommittee on Tests of Additions to Cement (H. J. Gilkey, chairman) has made a preliminary study of the data obtained in carrying out the prescribed tests of TDA, a material produced by the Dewey & Almy Chemical Co. of Cambridge, Mass., for use in portland cements of the type covered by the A.S.T.M. Standard Specifications C 9 – 38. The committee is not able at this time to make a final report.

Committee C-1 has approved the recommendations of the Sponsoring Committee on Masonry Cement (H. D. Baylor, chairman) that the Tentative Specifications for Masonry Cement (C 91 – 38 T)[8] be revised and adopted as standard. The revision, given below, consists of a change in the setting time requirements, together with an optional requirement and a method of test covering the propensities of this kind of cement for use in the staining of limestone:

Section 2.—Change the last sentence to read as follows by the addition of the italicized figure and the omission of that in brackets:

> Final set shall be attained within [48] *24* hr.

New Section.—Add as a new Section 6 an optional nonstaining requirement, to read as follows, renumbering the subsequent sections accordingly:

> 6. *Staining*.—This requirement shall apply only when the invitation for bids specifically states that the cement shall be nonstaining to limestone. Nonstaining cement shall contain not more than 0.03 per cent soluble "alkali" when tested in accordance with Section 22 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114) of the American Society for Testing Materials.
>
> NOTE.—The amount and nature of the staining material in limestones seem to vary with the stone. The alkali in any cement may, therefore, induce markedly different staining on different stone, even though the stone may have come apparently from the same source. The amount of alkali permitted by the specifications should not cause stain unless stone high in staining material has been used, or unless insufficient means have been used to prevent infiltration of water into the masonry.

Section 18.—Because of the revision in Section 2 omit the last sentence of Section 18 which reads as follows:

> If pats have not attained final set at the end of 24 hr., they shall remain in the moist cabinet for 48 hr. before being subjected to the steam test.

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[4] 1939 Book of A.S.T.M. Standards, Part II, p. 883.

[5] See p. 244.

[6] These methods were accepted as tentative by the Society and have been incorporated in Tentative Methods C 114 which appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 171.

[7] The procedures were adopted as standard by the Society and have been incorporated in the revised Standard Methods C 114 which appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 8.

[8] 1939 Book of A.S.T.M. Standards, Part II, p. 877.

Committee C-1 recommends that the following revisions be approved for immediate adoption in the Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 - 39):

Section 2.—Change Paragraph (*a*) from its present form to read as follows by the addition of the italicized words:

> (*a*) Samples for purpose of tests shall weigh at least 4 lb. *each when they are to be composited; single test samples, on which all specified tests are to be made, shall weigh at least 8 lb.*

Sections 2 (*b*), *3, 4* (*a*), *and* (*c*).—Change the number of barrels of cement represented by the test sample from "300 bbl." to read "400 bbl."

Section 4.—Combine with Section 3, relettering Paragraph (*b*) as (*c*) and change the second sentence of present Paragraph (*b*) from its present form: namely,

> Tubes inserted horizontally may be used where the construction of the storage permits.

to read as follows:

> This method may be used only when other methods cannot be applied, and great care must be exercised in order to obtain samples that completely represent the contents of the bin.

Section 5.—Designate this section as Paragraph 4 (*a*) and add a new Paragraph (*b*) to read as follows:

> (*b*) Composite samples for the tests as required in Section 6 shall be prepared by arranging all test samples in groups, each group representing the number of barrels required by the test or tests for which the composite is intended. From each of the test samples in a group, equal portions shall be taken, sufficient in amount to form a composite large enough to permit the making of the required physical or chemical determination. The composite sample thus prepared shall be thoroughly mixed before using.

New Section.—Add a new Section 5 to read as follows:

> 5. *Number of Physical and Chemical Tests.*—(*a*) All required physical tests shall be made on each test sample, taken as specified in Section 3, from trucks and cars, no test sample to represent more than one car.
>
> (*b*) Physical tests on samples taken from bins, boats, warehouses, etc., shall be required as follows:

Time of setting	each test sample representing each 400 bbl.
Fineness	composite sample representing each 800 bbl.
Strength	composite sample representing each 800 bbl.
Autoclave expansion	composite sample representing each 2000 bbl.

> (*c*) Chemical determinations shall be required as follows:

SO_3	composite sample representing each 800 bbl.
All specified chemical determinations (except SO_3)	composite sample representing each 4000 bbl.

> (*d*) When the total number of barrels sampled is less than that specified for any one of the above test or composite samples, all of the respective physical and chemical tests shall be made on the quantity sampled.

Section 10—Change the first sentence to read as follows by the addition of the italicized word:

> The quantities of dry materials to be mixed at one time shall be 500 g. for neat cement mixtures, and not less than 1000 nor more than 1200 g. for *briquet* mortar mixtures.

Figure 3.—Change the caption for Fig. 3 (*a*) to read as follows by the addition of the italicized words:

> (*a*) Shrinkage Cracks, *Shown Above,* Due to Exposure of Pats to Dry Air During Setting *Are not to Be Considered as Evidences of Unsoundness.*

The Working Committee on Fineness is not recommending any changes in the Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 - 38 T). Recently a method for determining the specific surface of cement by determining the permeability of a short column of cement to air was proposed abroad. Some studies by one of the members of the committee showed that the method has much promise. Less time is required for making the determination than when the Wagner turbidimeter is used. The method, however, does not give data covering size distribution.

The subcommittee on Cement Reference Laboratory (G. E. Warren, chairman), has submitted the following summary of its work for the period July 1, 1939, to June 30, 1940:

The Reference Laboratory continued the field work of the sixth inspection tour among cement laboratories. Approximately 300 laboratories are involved in that tour, which is now about 85 per cent completed. Additional equipment was secured to provide for field tests of certain apparatus not formerly included in the inspectors' routine work.

Reference Laboratory sample No. 3 was distributed and a summary of the results of tests thereon was sent to the 208 participating laboratories. Copies of the summary can be obtained upon request to the Cement Reference Laboratory at the National Bureau of Standards, Washington, D. C. The work included the usual physical and chemical tests. In addition, 49 laboratories reported heat-of-hydration values. The correspondence indicated great interets in the work and is serving to bring to attention many details of procedure. The agreement in chemical results was somewhat better for sample No. 3 than for No. 2. For sample No. 3 the agreement in chemical results was better for those laboratories which had participated in the tests of both samples Nos. 2 and 3 than for those laboratories which had tested No. 3 only.

The Reference Laboratory has practically completed the tests of TDA in standard portland cement, which tests were made for the Subcommittee on Tests of Additions to Cement.

The Reference Laboratory is cooperating with the subcommittees of Committee C-1 by making available to them the various suggestions and data accumulated during the course of the work.

The present officers of the committee were re-elected for the ensuing term of two years.

RECOMMENDATIONS AFFECTING STANDARDS

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 83 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Portland Cement (Effective September 2, 1940) . . .	66	4	7
Test for Autoclave Expansion of Portland Cement	72	1	4
II. REVISION OF TENTATIVE STANDARDS			
Chemical Analysis of Portland Cement (C 114 - 39 T): New quick method for determination of silicon dioxide, calcium oxide, and magnesium oxide	50	0	13
III. REVISIONS OF STANDARDS			
Sampling and Physical Testing of Portland Cement (C 77 - 39), immediate adoption	66	2	5
Chemical Analysis of Portland Cement (C 114 - 39), immediate adoption: New alternate method for determining phosphorus pentoxide, new method for determining chloroform-soluble organic substances . . .	67	0	6
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Masonry Cement (C 91 - 38 T): Change in setting time requirement, addition of optional nonstaining requirequirement and a test method therefor	67	0	6
Chemical Analysis of Portland Cement (C 114 - 39 T), Sections 1 to 9, as revised	69	0	6

This report has been submitted to letter ballot of the committee, which consists of 83 voting members; 63 members returned their ballots, of whom 58 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

P. H. BATES,
Chairman.

L. W. WALTER,
Secretary.

REPORT ON INVESTIGATION OF MORTARS BY SEVEN LABORATORIES

Sponsored by Working Committee on Plastic Mortar Tests

The Working Committee on Plastic Mortar Tests has completed an investigation of certain mortar mixes which supplements the series of tests sponsored by the committee in 1938.[1]

Purpose:

The investigation reported in 1938[1] showed that the so-called pebble mortars, made with sand and pebbles of three sizes and grading from minus No. 200 to $\frac{3}{8}$ in. with gap gradings, were better than the C 109 mortar[2] as to plasticity and water retention, and somewhat better in producing uniformity of test results but were more difficult to mix and to mold into satisfactory cubes. The purpose of the present series was to determine whether the addition of potter's flint to sands of the Ottawa type would produce a mortar having the satisfactory characteristics of the pebble mortars without the difficulties of mixing and placing; to determine whether a higher water-cement ratio, wetter consistency, and fixed proportions of cement, sand, and water would improve the pebble mortars, and whether local pebbles could be substituted for low-absorptive (0.4 per cent) pebbles without making a correction for the absorption of mixing water.

Materials:

The low-absorptive pebbles were purchased from a quarry in Columbia, S. C., and the potter's flint from a quarry in Ottawa, Ill. These sources of materials are the same as those in the 1938 series.[1] The standard and the graded Ottawa sand were furnished by each laboratory from its own supply, and the local pebbles by each laboratory from a local source. The cements were donated, proportioned, and distributed to the laboratories by the manufacturers which were as follows:

Aetna Portland Cement Co., Bay City, Mich.
Alpha Portland Cement Co., Easton, Pa.
Lehigh Portland Cement Co., Allentown, Pa.
Lone Star Cement Corp., New York, N. Y.
Universal Atlas Cement Co., New York, N. Y.

The following laboratories participated in the investigation:

Name	Individual
Lehigh Portland Cement Co., Allentown, Pa.	J. C. Pearson
Lone Star Cement Corp., Hudson, N. Y.	M. A. Swayze
National Bureau of Standards, Washington, D. C.	P. H. Bates
Portland Cement Assn., Chicago, Ill	H. F. Gonnerman
Public Roads Administration, Washington, D. C.	F. H. Jackson
E. W. Reed-Lewis, Detroit, Mich	E. W. Reed-Lewis
Universal Atlas Cement Co., New York, N. Y.	O. L. Moore

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 267 (1938).

[2] Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 - 37 T), 1939 Book of A.S.T.M. Standards, Part II, p. 892.

Description of Tests:

Compressive strength tests were made by six laboratories using six mixes and six cements, three standard (Nos. 1, 2, and 3) and three high-early-strength (Nos. 4, 5, and 6) portland cements, selected by E. W. Reed-Lewis as covering a wide range in "relative water capacity." The seventh laboratory did not make strength tests but investigated the "relative water capacity" of the cements using the methods of Mr. Reed-Lewis. Two of the six laboratories did this additional work also. These data have been submitted to Mr. Reed-Lewis for study and report.

The basic mix proportions used for 6-cube batches are given in Table I.

TABLE I.—BASIC MIX PROPORTIONS FOR 6-CUBE BATCHES.

Materials	Mix Designation					
	B-1	B-1L	B-2	B-3	B-4	C 109
Cement, g	450	450	460	500	440	470
Potter's flint, g	110	110	110	110	110	0
Standard Ottawa sand, g	790	790	790	0	1200	0
C 109 sand, graded Ottawa, g	0	0	0	1330	300	1300
Low-absorptive pebbles (⅜-in. to No. 4), g	1350	0	1350	0	0	0
Local pebbles (⅜-in. to No. 4), g	0	1350	0	0	0	0
Water, ml	239	239	266	265	234	250
c/w (cement-water ratio) by weight	1.88	1.88	1.73	1.88	1.88	1.88
w/c (water-cement ratio) by volume	0.80	0.80	0.87	0.80	0.80	0.80

The test specimens were 2-in. cubes, three for each period, and the test periods, 1, 3, 7, and 28 days. The tests were repeated, making a total of three complete rounds.

Referring to the basic mix proportions given in Table I, the mixes B-2 and C 109 were used as given, without any adjustment to control consistency, but the flow of the mortars was determined by the 10-in. flow table[3] and recorded. The other four mixes, B-1, B-1L, B-3, and B-4, were adjusted to give a flow within the range of 75 to 90 per cent by adding to or deducting from the basic mixes, when necessary, cement and water in the ratio of 1.88 by weight (water-cement ratio 0.8 by volume).

The cements were tested also in accordance with the following methods of the American Society for Testing Materials:

Chemical Analysis.—Standard Methods of Chemical Analysis of Portland Cement (C 114 - 39).[4]

Physical Tests.—Standard Methods of Sampling and Physical Testing of Portland Cement (C 77 - 37).[5]

Specific Surface.—Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 - 38 T).[6]

Three laboratories sent samples of the graded Ottawa sand used to the Cement Reference Laboratory for sieve analyses, which showed that the sands conformed to the requirements prescribed in Tentative Methods C 109 - 37 T.[2]

Discussion of Results:

The comments of the laboratory operators covering workability, ease of mixing, moldability, and water gain for each mix and each cement usually do not agree on any of the properties so that no single mortar can be selected as out-

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[3] Tentative Specifications for Masonry Cement (C 91 - 38 T), 1939 Book of A.S.T.M. Standards, Part II, p. 877.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 8.

[5] 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

[6] 1939 Book of A.S.T.M. Standards, Part II, p. 897.

TABLE II.—CHANGES IN BASIC MIX, AND FLOW.
Each value is average of three tests made on different days.

Laboratory	Change in Basic Mix Cement, g.						Flow, per cent					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
Cement No. 1												
No. 1	−75	−50	0	−50	−25	0	77	86	150	90	92	133
No. 2	0	+25	0	0	0	0	82	84	110	88	83	90
No. 3	−25	−25	0	−50	0	0	86	81	141	83	100	100
No. 4	0	0	0	0	0	0	87	86	114	81	79	87
No. 5[a]	+40S +68P	+40S +68P	+40S +68P	+67G	0	0	88	86	123	82	80	90
No. 6	−25	−25	0	−50	−25	0	96	90	129	88	86	118
Cement No. 2												
No. 1	−50	−25	0	−50	−25	0	84	84	140	90	87	133
No. 2	+25	+50	0	0	+25	0	83	84	104	81	83	82
No. 3	−25	−25	0	0	0	0	84	78	128	104	92	102
No. 4	+13	+13	0	0	0	0	82	84	107	81	79	85
No. 5	0	0	0	0	0	0	83	84	113	91	78	96
No. 6	−25	−25	0	−50	−25	0	81	86	120	84	83	122
Cement No. 3												
No. 1	−50	−25	0	−50	−25	0	88	88	143	87	87	131
No. 2	+25	+50	0	0	+25	0	81	82	98	78	82	81
No. 3	−25	−25	0	0	0	0	79	77	126	97	88	95
No. 4	0	0	0	0	0	0	79	79	103	81	77	87
No. 5	0	0	0	0	0	0	85	87	115	93	78	95
No. 6	−25	−25	0	−50	−25	0	79	76	118	77	78	109
Cement No. 4												
No. 1	−25	+25	0	−50	−25	0	77	84	121	89	79	124
No. 2	+75	+100	0	+25	+50	0	81	79	82	83	83	82
No. 3	0	+50	0	0	0	0	78	81	101	92	81	94
No. 4	+25	+50	0	0	+13	0	77	80	84	83	79	94
No. 5[a]	+45C +11F	+45C +11F	+45C +11F	+67G	0	0	79	78	90	81	76	93
No. 6	0	0	0	−25	−25	0	76	75	102	88	78	107
Cement No. 5												
No. 1	−25	+25	0	−75	−25	0	85	93	129	80	87	135
No. 2	+25	+50	0	0	+25	0	78	78	89	85	83	84
No. 3	0	0	0	0	+25	0	83	80	111	105	91	98
No. 4	+13	+13	0	−25	0	0	82	84	107	81	86	106
No. 5[a]	+40C +11F	+40C +11F	+40C +11F	+67G	0	0	88	84	99	83	77	101
No. 6	0	0	0	−50	0	0	79	78	108	84	87	113
Cement No. 6												
No. 1	0	+50	0	−50	−25	0	75	84	108	87	76	121
No. 2	+100	+150	0	+50	+75	0	78	81	71	85	84	79
No. 3	+100	+100	0	0	+25	0	85	85	96	89	80	93
No. 4	....	....	....	....	....	....	....	....	....	....	....	....
No. 5[a]	+112C +28F	+112C +28F	+112C +28F	0	+44C +11F	0	93	80	80	85	84	93
No. 6	+38	+25	0	0	0	0	78	75	90	90	79	103

[a] The abbreviations used in connection with the results from this laboratory indicate the following:
C = Cement.
F = Potter's flint.
S = Standard Ottawa sand.
G = Graded Ottawa sand.
P = Pebbles.

TABLE III.—COMPRESSIVE STRENGTH OF 2-IN. CUBES IN POUNDS PER SQUARE INCH.
Each value is average of nine cubes, three each made on different days.

Laboratory	1 Day						3 Days					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
Cement No. 1												
No. 1	744	740	542	413	408	339	2000	1950	1490	1230	1220	1050
No. 2	800	757	554	427	372	300	1960	1938	1540	1428	1225	1008
No. 3	830	830	585	450	495	415	1900	1985	1515	1220	1365	1160
No. 4	683	619	450	392	389	297	1733	1706	1292	1245	1225	906
No. 5	735	715	490	405	430	310	1975	2100	1575	1305	1370	1050
No. 6	597	667	430	383	397	307	1613	1587	1403	957	1013	797
Average	727	721	509	412	415	328	1864	1878	1469	1231	1236	995
Percentage mean variation	8.6	7.5	10.2	4.8	7.6	9.9	6.8	8.2	5.5	7.7	7.1	9.6
Cement No. 2												
No. 1	933	884	686	535	555	488	2630	2540	2030	1690	1720	1540
No. 2	987	1057	772	596	650	490	2789	2668	2055	1961	2035	1592
No. 3	1070	1145	775	760	720	605	2775	2810	2155	2390	2100	1825
No. 4	1042	922	725	600	583	489	2570	2472	2000	2017	1895	1514
No. 5	900	865	665	625	590	470	2640	2630	2025	2020	1865	1585
No. 6	707	763	527	450	457	377	2150	2400	1620	1330	1403	1180
Average	979	939	692	594	593	486	2592	2587	1981	1901	1836	1539
Percentage mean variation	9.5	11.4	9.5	11.5	10.5	8.7	6.0	4.5	6.1	12.7	9.1	8.3
Cement No. 3												
No. 1	816	816	610	531	533	427	2360	2380	1850	1590	1540	1390
No. 2	843	913	667	558	579	435	2350	2535	1875	1606	1754	1385
No. 3	1090	1100	790	835	785	630	2605	2600	2015	1965	1910	1635
No. 4	856	833	583	544	533	408	2264	2303	1725	1606	1536	1353
No. 5	815	860	615	530	610	410	2435	2510	1970	1655	1830	1390
No. 6	647	767	480	453	463	353	2025	2307	1547	1290	1373	1137
Average	845	882	624	575	584	444	2340	2439	1997	1619	1657	1382
Percentage mean variation	10.2	9.5	11.1	15.0	13.0	14.0	5.6	4.4	8.6	7.7	10.5	6 6
Cement No. 4												
No. 1	2490	2290	1860	1760	1800	1580	4890	4870	3970	3470	3450	3380
No. 2	2200	2192	1872	1978	1857	1586	4589	4538	3815	4047	3839	3224
No. 3	2555	2345	2120	2200	2100	1840	5030	4965	4330	4455	4210	3680
No. 4	2230	2225	1767	1978	1917	1492	4650	4536	3750	3986	3861	3097
No. 5	2305	2380	1825	1895	1780	1515	4810	4960	3900	3800	3560	3215
No. 6	1943	2097	1460	1527	1513	1263	4813	5160	3890	3883	3733	3233
Average	2287	2255	1817	1890	1828	1546	4797	4838	3943	3940	3776	3305
Percentage mean variation	6.4	3.7	7.5	8.6	7.1	8.1	2.5	4.2	3.5	5.7	5.2	4.5
Cement No. 5												
No. 1	2040	1940	1620	1460	1440	1380	4170	4260	3360	2920	3000	3040
No. 2	2048	2015	1550	1544	1244	1075	4150	4254	3478	3360	3249	2807
No. 3	1985	2055	1590	1755	1760	1435	4295	4340	3595	3895	3865	3305
No. 4	1889	1906	1406	1634	1353	1217	4205	4175	3253	3439	3186	2825
No. 5	1850	1940	1445	1575	1505	1360	4175	4310	3380	3515	3320	3010
No. 6	1657	1747	1207	1210	1233	1083	4553	4970	3463	3330	3453	2977
Average	1912	1927	1470	1530	1423	1258	4258	4385	3422	3410	3345	2977
Percentage mean variation	5.9	3.8	8.0	8.5	10.2	10.6	2.6	4.5	2.6	6.1	6.2	4.2
Cement No. 6												
No. 1	1960	1840	1500	1550	1480	1340	4840	4670	4120	3710	3670	3360
No. 2	1858	1822	1487	1667	1560	1370	4507	4465	3749	3931	3823	3346
No. 3	2150	2275	1830	2090	2010	1745	4550	4835	4060	4150	4210	3540
No. 4	....	....	....	....	....	....	....	....	....	....	....	....
No. 5	1970	1980	1550	1715	1675	1350	4850	4825	4030	4225	4095	3660
No. 6	1760	2003	1423	1533	1457	1237	4823	5333	3970	4113	3807	3390
Average	1940	1984	1558	1711	1636	1408	4714	4826	3986	4026	3921	3459
Percentage mean variation	5.4	6.2	7.0	7.8	10.1	9.5	2.3	4.3	2.5	4.1	4.7	3.3

TABLE III.—COMPRESSIVE STRENGTH OF 2-IN. CUBES IN POUNDS PER SQUARE INCH (*Continued*).

Laboratory	7 Days						28 Days					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
Cement No. 1												
No. 1	3170	3080	2240	1920	1920	1680	6680	6560	5830	4370	4590	4290
No. 2	3029	3057	2265	2336	1982	1771	6318	6236	5268	5296	4457	4254
No. 3	3005	3185	2370	2030	2380	1925	6295	6295	5405	4075	4845	4345
No. 4	2867	2811	2256	2128	2028	1461	6806	6941	5539	4867	4667	3892
No. 5	3335	3365	2505	2120	2165	1665	7030	7315	5925	4820	5060	4225
No. 6	3073	3443	1757	1577	1653	1360	7140	7650	5663	4650	5100	4380
Average	3079	3157	2232	2019	2021	1644	6712	6833	5605	4680	4786	4231
Percentage mean variation	3.7	5.6	7.1	8.9	7.5	9.4	4.2	6.9	3.6	6.7	4.5	2.3
Cement No. 2												
No. 1	4690	4570	3820	2990	3100	2950	7520	7590	7020	5050	5190	5240
No. 2	4679	4674	3725	3450	3872	3076	7474	7177	6411	5878	6130	5443
No. 3	4765	4605	3840	4095	3540	3270	7440	6995	6755	6315	5600	5205
No. 4	4845	4889	3961	3806	3381	3067	7689	8117	6622	6325	5869	5153
No. 5	4780	4650	3895	3785	3385	3010	7635	7625	7020	6285	5865	5345
No. 6	4663	5180	3923	3190	3230	3040	7785	8355	7230	5525	5750	5605
Average	4737	4761	3861	3553	3418	3069	7591	7643	6843	5896	5734	5332
Percentage mean variation	1.3	3.8	1.7	9.7	5.6	2.3	1.5	5.2	2.9	7.0	3.9	2.5
Cement No. 3												
No. 1	3930	4080	3440	2560	2600	2430	6160	6440	6060	4170	4390	4420
No. 2	3717	3939	3439	2944	3043	2482	6079	6531	5281	4577	4639	4017
No. 3	3960	4045	3470	3395	3140	2640	6065	6070	5865	5250	4935	4440
No. 4	4147	4053	3128	2753	2694	2294	6528	6375	5614	4542	4756	4064
No. 5	4260	4325	3635	2920	3240	2485	6735	7020	6260	4795	5205	4330
No. 6	4110	4757	3307	2717	2860	2553	7125	7680	6055	4640	4925	4660
Average	4021	4200	3403	2882	2928	2481	6449	6686	5856	4662	4808	4322
Percentage mean variation	3.7	5.4	3.6	6.5	7.2	3.1	5.4	6.6	4.6	5.2	4.4	4.3
Cement No. 4												
No. 1	6520	6590	5600	4910	4760	4510	8120	8390	7230	6120	6010	6100
No. 2	6175	6045	5404	5361	5298	4540	7464	7437	6885	6806	6342	5710
No. 3	6400	6975	6000	5905	5305	5035	7975	8020	7315	6985	6350	6295
No. 4	6636	6589	5708	5461	5416	4369	8158	7955	6986	6983	6314	6005
No. 5	6855	7085	5885	5270	5075	4745	8500	8960	7565	6810	6465	6350
No. 6	6605	6880	5485	5195	4995	4730	8295	8290	7345	6680	6415	6170
Average	6532	6694	5680	5350	5142	4655	8084	8175	7221	6731	6316	6105
Percentage mean variation	2.5	4.3	3.2	4.3	4.3	3.9	3.0	4.5	2.6	3.3	1.6	2.7
Cement No 5												
No. 1	6870	6770	6140	4830	4890	5200	8700	8380	7980	6200	6690	6880
No. 2	6699	6451	5720	5564	5315	4906	7989	7974	7500	7065	6731	6489
No. 3	6400	6000	5590	6115	5940	5175	7710	7365	7240	7520	6965	6765
No. 4	6964	7061	5661	5586	5227	5197	8350	8247	7442	7022	6681	6222
No. 5	6865	6995	6010	5780	5525	5425	8640	8770	7790	7335	7100	7240
No. 6	7135	7270	5810	5385	5500	5300	8870	9030	7645	7040	7335	7010
Average	6822	6758	5822	5543	5399	5201	8377	8294	7599	7030	6917	6768
Percentage mean variation	2.7	5.2	2.9	4.6	4.7	2.1	4.3	5.2	2.7	3.7	3.5	4.1
Cement No. 6												
No. 1	6790	6600	5730	4920	4990	4920	8590	8630	7610	6560	6560	6490
No. 2	6021	5978	5164	5535	5368	4574	7865	7650	7086	7125	6868	6236
No. 3	6400	6580	5665	5400	5475	5065	7815	8215	7535	6715	6695	6450
No. 4	....	....	....	....	....	....	....	....	....	....	....	....
No. 5	6520	6680	5655	5735	5570	5145	8430	8540	7595	7415	7290	6705
No. 6	6555	7155	5695	5695	5215	4775	8635	9400	7765	7405	7057	6607
Average	6457	6599	5582	5457	5324	4896	8267	8487	7518	7044	6894	6500
Percentage mean variation	3.0	3.9	3.0	4.4	2.9	3.6	4.1	5.2	2.3	4.6	3.3	2.0

TABLE IV.—PERCENTAGE MEAN VARIATION IN COMPRESSIVE STRENGTH OF THREE TESTS FROM THEIR AVERAGE.

Tests made on three days.

Laboratory	1 Day						3 Days					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
CEMENT NO. 1												
No. 1	6.3	2.4	3.9	3.4	2.7	2.9	1.8	1.9	0.9	1.1	0.8	1.6
No. 2	8.7	5.0	6.5	5.1	10.0	1.7	7.4	2.9	2.0	5.1	6.9	6.3
No. 3	5.2	3.4	4.3	11.1	12.1	9.2	4.7	2.8	5.0	7.6	4.5	4.7
No. 4	18.1	11.0	6.2	2.8	3.3	5.0	3.5	4.4	6.9	5.5	1.8	2.8
No. 5	11.1	6.7	4.5	2.0	4.2	3.2	4.1	4.9	4.0	0.7	1.8	1.4
No. 6	9.7	5.6	6.2	4.1	5.6	9.5	14.0	18.6	12.5	1.6	1.7	2.0
Average	9.9	5.7	5.3	4.8	6.3	5.3	5.9	5.9	5.2	3.6	2.9	3.1
CEMENT NO. 2												
No. 1	1.5	1.2	2.8	0.6	1.4	0.6	0.1	0.3	2.6	0.8	1.0	1.5
No. 2	9.0	3.6	4.1	5.6	9.8	8.1	2.5	6.9	9.5	3.2	7.4	7.2
No. 3	3.6	8.0	4.9	2.6	2.5	0.8	5.4	3.4	3.9	0.9	3.2	5.2
No. 4	8.0	11.9	2.3	5.6	8.6	11.7	2.1	13.9	1.4	2.8	7.1	2.7
No. 5	3.6	2.0	3.8	1.9	2.0	6.8	1.7	4.3	1.4	3.2	1.2	4.2
No. 6	1.3	3.8	4.8	0.4	3.9	4.2	9.9	8.3	11.1	3.5	1.3	3.4
Average	4.5	5.1	3.8	2.8	4.7	5.4	3.6	6.2	5.0	2.4	3.6	4.0
CEMENT NO. 3												
No. 1	1.0	2.9	4.6	4.0	3.4	4.0	1.6	1.7	0.9	1.3	1.5	0.7
No. 2	5.4	4.6	3.3	4.5	7.2	3.7	4.8	1.8	0.6	3.7	3.6	0.7
No. 3	3.9	2.5	1.0	5.4	4.5	6.4	3.5	2.7	1.4	3.7	0.9	2.8
No. 4	6.9	8.7	6.7	4.5	2.1	8.2	6.2	5.4	6.1	2.3	3.9	10.4
No. 5	3.9	4.9	2.9	13.8	14.4	2.4	3.9	5.5	4.2	11.3	9.7	0.6
No. 6	4.5	1.6	5.6	1.0	2.4	5.0	9.8	10.8	9.2	1.6	0.8	2.7
Average	4.3	4.2	4.0	5.5	5.7	5.0	5.0	4.7	3.7	4.0	3.4	3.0
CEMENT NO. 4												
No. 1	1.3	1.0	1.6	2.4	2.9	1.9	1.4	1.2	2.3	5.5	2.6	1.9
No. 2	6.2	5.3	5.1	2.4	1.6	3.6	6.3	3.9	3.8	1.2	1.9	2.9
No. 3	2.0	8.5	6.4	7.4	9.0	7.7	4.6	0.8	1.3	5.8	3.7	1.8
No. 4	4.6	1.5	5.3	5.8	7.5	7.5	2.8	6.1	3.3	6.7	9.0	6.3
No. 5	2.0	1.8	3.4	2.7	1.3	2.5	1.0	2.0	0.7	1.5	1.9	5.0
No. 6	2.6	3.9	0.9	2.0	3.4	3.0	2.7	2.1	3.9	1.1	2.4	1.7
Average	3.1	3.7	3.8	3.8	4.3	4.4	3.1	2.7	2.6	3.6	3.6	3.3
CEMENT NO. 5												
No. 1	0.5	1.7	3.3	1.8	1.6	0.5	1.0	3.2	1.2	0.8	2.8	1.1
No. 2	6.9	1.1	6.1	5.3	20.7	21.2	7.3	7.3	7.2	8.3	3.7	4.3
No. 3	3.4	8.9	8.3	5.1	3.4	5.6	2.9	6.7	1.5	6.0	2.0	0.9
No. 4	1.7	2.8	2.9	2.7	1.4	4.6	1.6	4.1	5.6	3.1	2.6	1.2
No. 5	5.1	5.2	3.9	3.9	2.8	2.8	3.2	1.6	3.8	0.9	3.4	0.6
No. 6	2.6	2.9	3.5	2.2	1.3	2.2	2.7	1.2	2.9	1.8	1.9	3.2
Average	3.4	3.8	4.7	3.5	5.2	6.2	3.1	4.0	3.7	3.5	2.7	1.9
CEMENT NO. 6												
No. 1	1.2	0.5	1.3	2.6	3.4	2.0	1.3	1.5	1.7	0.8	1.9	0.5
No. 2	2.1	3.6	4.1	3.8	2.4	10.2	3.5	3.7	5.0	5.3	3.8	2.8
No. 3	1.6	1.0	4.3	3.4	2.7	7.0	5.2	7.2	1.3	2.6	1.3	2.2
No. 4	....	....	....	....	....	....	....	....	....	....	....	....
No. 5	5.0	3.6	5.5	5.1	2.5	6.6	3.4	3.2	3.8	0.4	1.1	1.6
No. 6	2.3	1.4	1.2	2.3	2.6	3.6	2.1	1.4	1.7	1.5	1.7	3.5
Average	2.4	2.0	3.3	3.4	2.7	5.9	3.1	3.4	2.7	2.1	2.0	2.1

TABLE IV.—PERCENTAGE MEAN VARIATION IN COMPRESSIVE STRENGTH OF THREE TESTS FROM THEIR AVERAGE (*Continued*).

Laboratory	7 Days						28 Days					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
CEMENT NO. 1												
No. 1	3.2	3.8	1.3	0.5	2.6	2.6	0.0	2.1	1.1	1.0	0.3	1.4
No. 2	5.4	2.2	2.1	5.5	7.8	11.5	6.8	1.9	6.4	4.6	3.5	4.4
No. 3	4.6	3.9	4.6	4.2	8.7	0.5	2.4	2.8	1.2	0.7	3.0	1.5
No. 4	3.7	1.4	5.1	6.2	3.2	2.1	4.0	1.4	4.0	5.7	2.5	10.1
No. 5	6.0	1.4	3.2	1.3	1.4	3.9	2.8	0.8	4.2	1.3	1.9	4.1
No 6	2.7	2.4	0.6	4.1	2.7	0.5	0.6	1.2	0.3	3.9	1.4	0.7
Average	4.3	2.5	2.8	3.6	4.4	3.5	2.8	1.7	2.9	2.9	2.1	3.7
CEMENT NO. 2												
No. 1	1.5	0.7	2.3	2.6	0.5	0.7	1.1	1.6	1.3	1.0	0.8	0.5
No. 2	5.8	4.8	9.7	4.1	6.3	5.4	3.2	0.4	1.0	3.7	5.9	3.4
No. 3	1.8	3.5	2.8	0.5	6.9	2.2	2.6	9.1	1.6	3.8	2.7	7.0
No. 4	5.1	6.3	4.6	6.0	7.1	4.2	1.6	1.4	2.3	2.8	3.8	5.0
No. 5	1.4	5.4	2.1	2.7	0.5	2.8	1.1	1.5	3.2	4.0	2.6	3.2
No. 6	0.3	1.6	5.9	2.3	1.7	3.5	0.1	2.6	8.0	0.6	2.3	1.3
Average	2.7	3.7	4.6	3.0	3.8	3.1	1.6	2.8	2.9	2.7	3.0	3.5
CEMENT NO. 3												
No. 1	1.1	3.4	1.7	2.7	1.4	0.7	1.7	0.5	0.9	2.6	2.5	1.7
No. 2	1.3	6.2	4.3	3.3	2.0	2.5	5.4	5.0	2.7	3.5	0.2	1.4
No 3	3.0	2.3	1.6	2.0	2.6	1.0	2.4	6.2	0.7	1.9	1.6	2.6
No. 4	1.1	2.9	2.9	5.1	4.6	4.9	4.7	7.3	7.5	1.3	2.0	4.4
No. 5	2.5	3.0	2.7	8.4	8.8	1.0	1.5	1.3	3.2	8.8	6.6	1.9
No. 6	4.2	2.4	2.5	3.9	1.9	4.3	3.0	4.1	3.2	1.5	1.9	4.1
Average	2.2	3.4	2.6	4.2	3.6	2.4	3.1	4.1	3.0	3.3	2.5	2.7
CEMENT NO. 4												
No. 1	1.0	1.2	1.5	1.6	0.8	2.4	1.3	1.2	0.9	1.0	0.2	0.5
No. 2	4.9	6.0	2.7	5.5	3.6	5.6	3.7	0.8	1.8	0.8	3.6	2.4
No. 3	0.7	1.2	1.4	1.0	1.0	3.6	4.0	5.1	1.1	2.0	0.3	2.0
No. 4	0.5	2.7	2.0	3.7	5.7	3.3	3.6	4.2	0.7	4.7	3.1	1.6
No. 5	2.2	1.7	2.4	2.7	2.4	3.6	1.1	1.5	1.1	1.4	0.6	3.7
No. 6	0.7	1.0	1.0	0.3	0.9	1.9	3.2	1.0	2.7	3.3	2.9	3.9
Average	1.7	2.3	1.8	2.5	2.4	3.4	2.8	2.3	1.4	2.2	1.8	2.4
CEMENT NO. 5												
No. 1	1.1	2.1	1.6	1.6	4.1	0.6	2.1	1.6	0.5	1.6	1.0	0.6
No. 2	1.5	6.9	7.0	2.7	2.7	5.6	5.5	0.8	1.0	3.8	2.1	1.8
No. 3	1.5	11.4	2.2	1.6	3.1	4.9	2.2	4.5	1.4	1.3	1.4	1.4
No. 4	2.2	1.5	2.2	4.1	1.8	2.3	2.4	4.8	0.8	3.6	6.6	2.8
No. 5	3.8	4.3	2.4	1.3	2.8	3.6	2.2	1.8	0.9	0.2	1.6	2.0
No. 6	0.8	0.0	2.4	0.1	1.2	5.7	1.0	0.7	1.4	4 1	4.2	4.5
Average	1.8	4.4	3.0	1.9	2 6	3.8	2.6	2.4	1.0	2.4	2.8	2.2
CEMENT NO. 6												
No. 1	1.1	0.8	2.4	2.8	1.5	1.6	0.3	0.3	0.1	1.5	2.3	0.7
No. 2	2.7	2.7	3.3	2.1	1.8	4.8	0.6	3.3	1.0	1.4	0.9	1.4
No. 3	2.4	1.6	1.8	3.2	0.9	4.1	3.0	1.4	1.3	3.7	1.5	1.1
No. 4	....	....	....	....	....	....	....	....	....	....	....	....
No. 5	1.2	3.6	2.7	1.2	0.9	1.9	2.4	2.4	2.8	1.9	4.1	1.6
No. 6	1.1	0.6	0.6	2.5	0.4	0.5	1.0	1.9	3.9	0.7	2.0	0.4
Average	1.7	1.8	2.2	2.4	1.3	2.6	1.5	1.9	1.8	1.8	2.2	1.0

TABLE V.—PERCENTAGE MEAN VARIATION IN COMPRESSIVE STRENGTH OF THREE TEST SPECIMENS FROM THEIR AVERAGE.

Each value is average percentage mean variation of three tests made on three days.

Laboratory	1 Day						3 Days					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
Cement No. 1												
No. 1.........	1.8	3.7	3.3	0.6	1.2	2.3	2.9	2.2	2.8	2.4	0.9	2.3
No. 2.........	4.0	1.9	5.2	3.4	2.9	3.7	2.1	3.5	3.7	3.5	2.0	2.0
No. 3.........	2.2	3.5	3.0	4.1	2.4	2.0	1.9	3.8	3.6	2.7	2.4	1.1
No. 4.........	0.9	3.1	2.9	5.6	3.3	1.2	1.5	2.6	2.4	2.8	2.6	3.9
No. 5.........	5.8	3.8	5.0	1.9	2.3	2.6	4.9	2.8	3.3	1.6	2.2	2.1
No. 6.........	3.0	2.0	1.6	0.9	2.3	1.9	5.4	1.3	1.4	0.9	2.0	2.2
Average....	3.0	3.0	3.5	2.8	2.4	2.3	3.1	2.7	2.9	2.3	2.0	2.3
Cement No. 2												
No. 1.........	2.6	2.2	4.3	2.1	1.5	3.4	2.0	1.0	1.8	1.7	1.7	1.9
No. 2.........	1.5	1.9	2.2	1.4	2.5	4.1	1.5	1.6	1.8	2.9	2.6	3.0
No. 3.........	7.2	3.5	2.8	1.2	2.5	2.7	2.3	3.3	1.1	2.2	1.2	1.6
No. 4.........	1.8	3.3	2.3	0.9	3.0	1.8	1.3	2.3	2.8	1.8	2.0	3.4
No. 5.........	3.0	3.3	2.5	2.1	2.7	4.1	2.6	4.2	2.7	2.1	2.1	2.8
No. 6.........	1.9	2.5	1.5	1.0	2.0	1.5	0.8	1.8	1.0	1.3	1.3	3.1
Average....	3.0	2.8	2.6	1.5	2.4	2.9	1.8	2.4	1.9	2.0	1.8	2.6
Cement No. 3												
No. 1.........	2.3	1.0	3.5	0.8	1.4	1.0	0.9	1.6	2.0	1.9	1.3	2.5
No. 2.........	1.7	3.3	2.8	2.1	3.2	2.2	1.6	2.4	1.2	1.7	2.4	2.1
No. 3.........	3.8	5.3	2.6	3.0	2.2	1.9	1.5	1.8	1.4	2.8	3.0	3.0
No. 4.........	4.1	4.0	3.3	2.0	1.0	2.7	1.3	3.3	1.2	3.2	2.0	2.6
No. 5.........	3.4	2.0	2.3	2.8	1.3	2.8	2.9	2.4	2.4	1.9	0.8	2.1
No. 6.........	2.4	2.0	2.1	1.7	1.0	0.3	1.3	1.5	0.7	1.0	1.0	0.9
Average....	3.0	2.9	2.8	2.1	1.7	1.8	1.6	2.2	1.5	2.1	1.8	2.2
Cement No. 4												
No. 1.........	1.1	0.9	1.4	1.8	1.7	2.0	1.1	2.2	1.1	1.8	4.2	2.3
No. 2.........	2.3	4.0	2.1	2.2	1.8	1.9	2.2	2.4	1.3	1.5	1.8	1.9
No. 3.........	3.3	6.0	2.0	2.5	1.9	1.6	2.7	2.5	2.0	3.3	2.5	2.2
No. 4.........	1.8	2.0	2.3	2.4	1.2	3.6	0.7	0.8	1.3	1.8	1.2	1.0
No. 5.........	2.7	2.3	2.5	2.7	2.8	1.8	1.4	2.4	3.1	2.5	2.6	0.7
No. 6.........	1.7	1.9	1.2	0.9	1.3	2.2	0.3	0.7	0.8	0.9	1.0	0.7
Average....	2.1	2.8	1.9	2.1	1.8	2.2	1.4	1.8	1.6	2.0	2.2	1.5
Cement No. 5												
No. 1.........	0.8	1.5	1.5	1.7	1.9	1.2	1.3	2.8	1.9	1.5	2.3	1.6
No. 2.........	1.5	2.3	2.0	4.3	1.2	1.4	2.1	3.0	1.7	1.7	2.3	2.5
No. 3.........	1.5	2.1	1.4	2.2	1.3	4.1	3.0	3.7	2.3	4.1	1.5	1.6
No. 4.........	2.5	2.6	1.4	1.8	0.7	1.9	1.8	2.0	1.2	2.2	2.0	2.0
No. 5.........	2.4	3.5	2.6	0.9	2.2	2.0	2.5	2.3	2.0	2.4	1.7	1.3
No. 6.........	2.0	1.3	1.5	1.6	0.3	1.5	1.2	1.1	0.6	0.9	0.9	1.3
Average....	1.8	2.2	1.7	2.1	1.3	2.0	2.0	2.5	1.6	2.1	1.8	1.7
Cement No. 6												
No. 1.........	1.3	2.3	2.1	1.0	1.6	0.6	2.3	1.8	1.8	0.9	3.3	1.4
No. 2.........	1.4	1.4	2.1	1.6	1.5	0.8	1.3	2.1	1.3	0.8	1.8	1.3
No. 3.........	3.4	3.0	2.0	2.3	1.5	1.1	1.7	3.6	2.3	1.7	0.7	4.8
No. 4.........	....	....	....	....	....	....	....	....	....	....	....	....
No. 5.........	1.2	5.0	1.5	1.5	1.3	3.9	1.5	3.4	1.1	1.1	1.1	2.7
No. 6.........	1.0	1.3	0.8	0.8	1.0	1.4	0.4	1.1	1.4	0.4	1.0	1.4
Average....	1.7	2.6	1.7	1.4	1.4	1.6	1.4	2.4	1.6	1.0	1.6	2.3

TABLE V.—PERCENTAGE MEAN VARIATION IN COMPRESSIVE STRENGTH OF THREE TEST SPECIMENS FROM THEIR AVERAGE (*Continued*).

Each value is average percentage mean variation of three tests made on three days.

Laboratory	7 Days						28 Days					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
						CEMENT NO. 1						
No. 1	1.9	1.3	0.8	1.7	3.4	1.9	2.5	2.8	1.4	1.9	1.8	1.9
No. 2	2.0	3.1	2.2	1.9	2.0	2.0	3.4	2.6	3.2	2.9	3.7	4.3
No. 3	2.9	3.2	3.3	1.7	2.3	2.7	3.9	3.2	2.9	2.3	4.2	1.7
No. 4	1.2	1.1	1.0	1.9	1.5	2.8	1.5	1.3	2.0	1.0	1.7	3.7
No. 5	5.1	1.9	3.4	1.9	3.9	3.4	5.3	2.1	2.9	2.2	1.2	2.8
No 6	2.6	0.8	1.6	1.0	1.9	2.1	0.7	3.2	1.4	1.1	1.9	1.5
Average	2.6	1.9	2.1	1.7	2.5	2.5	2.9	2.5	2.3	1.9	2.4	2.6
						CEMENT NO. 2						
No. 1	1.8	2.8	0.7	3.7	1.2	1.6	2.3	3.3	1.5	3.1	3.6	3.7
No. 2	2.7	3.0	2.3	2.2	1.9	2.0	1.4	1.2	1.5	3.0	2.6	3.7
No. 3	2.5	5.0	2.1	1.2	3.9	3.7	3.0	5.8	2.4	2.8	1.2	2.2
No. 4	2.3	1.5	1.0	3.2	1.9	1.2	2.0	1.9	1.9	1.6	1.9	5.1
No. 5	2.3	1.3	2.4	1.8	1.5	2.9	1.6	2.8	1.1	3.0	2.7	2.5
No 6	1.0	1.1	0.7	0.9	1.3	1.5	1.4	2.0	0.7	0.5	0.8	0.6
Average	2.1	2.5	1.5	2.2	2.0	2.2	2.0	2.8	1.5	2.3	2.1	3.0
						CEMENT NO. 3						
No. 1	2.0	2.2	2.5	2.3	2.1	1.0	1.4	1.4	2.3	1.2	2.3	1.0
No. 2	3.1	6.2	1.6	4.2	2.8	2.8	1.4	2.6	1.9	4.0	3.9	3.2
No. 3	3.3	6.2	2.4	1.5	1.3	3.2	4.9	4.8	3.2	2.5	1.7	4.6
No. 4	0.8	2.0	1.2	3.4	2.2	2.1	1.6	1.9	1.2	4.0	2.2	2.8
No. 5	2.7	1.6	2.7	1.0	2.9	1.3	1.5	1.9	2.5	1.3	2.2	3.2
No. 6	0.9	1.5	0.7	1.5	1.0	1.1	1.1	1.3	1.2	0.9	0.6	0.7
Average	2.1	3.3	1.9	2.3	2.1	1.9	2.0	2.3	2.1	2.3	2.2	2.6
						CEMENT NO. 4						
No. 1	2.5	1.3	1.0	1.9	2.6	1.9	1.5	1.2	2.3	2.5	2.6	2.4
No. 2	3.6	1.0	2.4	3.4	1.3	2.7	1.1	2.1	2.6	2.5	4.1	2.5
No. 3	2.5	2.9	2.2	2.3	3.5	3.4	3.7	5.0	1.3	1.9	2.8	2.2
No. 4	1.7	1.1	0.9	0.9	2.5	2.5	1.7	3.3	1.6	1.5	2.7	2.3
No. 5	1.6	3.0	1.8	1.3	2.5	1.9	2.4	2.2	1.1	1.8	2.0	2.3
No. 6	1.3	0.1	0.4	0.4	0.8	0.4	1.0	1.2	0.7	1.1	1.0	1.2
Average	2.2	1.6	1.4	1.7	2.2	2.1	1.9	2.5	1.6	1.9	2.5	2.1
						CEMENT NO. 5						
No. 1	2.2	3.1	2.6	0.7	2.5	2.1	1.0	1.7	1.3	2.7	2.6	1.7
No. 2	1.3	1.9	2.5	3.0	1.6	2.9	2.6	1.5	1.4	4.2	1.4	1.6
No. 3	3.0	8.6	1.0	3.0	1.7	1.5	3.8	5.2	2.0	1.9	4.1	1.1
No. 4	0.9	1.1	1.6	2.3	3.6	1.8	2.2	1.7	1.1	0.6	1.4	2.1
No. 5	1.2	1.4	1.9	2.1	2.1	2.7	1.3	2.8	1.6	2.3	2.6	2.5
No. 6	1.5	1.7	0.7	0.5	1.1	1.6	1.4	3.3	1.1	0.5	0.9	1.8
Average	1.7	3.0	1.7	1.9	2.1	2.1	2.1	2.7	1.4	2.0	2.2	1.8
						CEMENT NO. 6						
No. 1	2.7	1.7	2.9	2.1	1.3	2.0	1.6	3.5	1.9	2.0	1.6	2.4
No. 2	1.9	2.1	2.9	2.3	0.9	1.5	1.7	2.1	1.4	1.5	1.9	1.3
No. 3	1.2	2.9	3.0	2.6	1.1	1.3	2.0	4.4	2.1	1.6	2.1	1.4
No. 4	....	....	....	....	....	....	....	....	....	....	....	..
No. 5	4.5	3.5	1.4	1.2	1.8	1.3	2.4	1.7	2.1	1.8	1.0	2.8
No. 6	1.4	0.7	0.5	0.7	0.5	0.9	1.2	1.4	0.5	1.0	0.5	0.5
Average	2.3	2.2	2.1	1.8	1.1	1.4	1.8	2.6	1.6	1.6	1.4	1.7

TABLE VI.—SUMMARY OF PERCENTAGE MEAN VARIATIONS.

Cement	1 Day						3 Days					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
Percentage Mean Variations of Compressive Strength between Laboratories (Values from Table III).												
No. 1	8.6	7.5	10.2	4.8	7.6	9.9	6.8	8.2	5.5	7.7	7.1	9.6
No. 2	9.5	11.4	9.5	11.5	10.5	8.7	6.0	4.5	6.1	12.7	9.1	8.3
No. 3	10.2	9.5	11.1	15.0	13.0	14.0	5.6	4.4	8.6	7.7	10.5	6.6
No. 4	6.4	3.7	7.5	8.6	7.1	8.1	2.5	4.2	3.5	5.7	5.2	4.5
No. 5	5.9	3.8	8.0	8.5	10.2	10.6	2.6	4.5	2.6	6.1	6.2	4.2
No. 6	5.4	6.2	7.0	7.8	10.1	9.5	2.3	4.3	2.5	4.1	4.7	3.3
Average	7.7	7.0	8.9	9.4	9.8	10.1	4.3	5.0	4.8	7.3	7.1	6.1
Average Percentage Mean Variations in Compressive Strength of Three Tests from Their Average (Values from Table IV).												
No. 1	9.9	5.7	5.3	4.8	6.3	5.3	5.9	5.9	5.2	3.6	2.9	3.1
No. 2	4.5	5.1	3.8	2.8	4.7	5.4	3.6	6.2	5.0	2.4	3.6	4.0
No. 3	4.3	4.2	4.0	5.5	5.7	5.0	5.0	4.7	3.7	4.0	3.4	3.0
No. 4	3.1	3.7	3.8	3.8	4.3	4.4	3.1	2.7	2.6	3.6	3.6	3.3
No. 5	3.4	3.8	4.7	3.5	5.2	6.2	3.1	4.0	3.7	3.5	2.7	1.9
No. 6	2.4	2.0	3.3	3.4	2.7	5.9	3.1	3.4	2.7	2.1	2.0	2.1
Average	4.6	4.1	4.2	4.0	4.8	5.4	4.0	4.5	3.8	3.2	3.0	2.9
Average Percentage Mean Variations in Compressive Strength of Three Test Specimens from Their Average (Values from Table V).												
No. 1	3.0	3.0	3.5	2.8	2.4	2.3	3.1	2.7	2.9	2.3	2.0	2.3
No. 2	3.0	2.8	2.6	1.5	2.4	2.9	1.8	2.4	1.9	2.0	1.8	2.6
No. 3	3.0	2.9	2.8	2.1	1.7	1.8	1.6	2.2	1.5	2.1	1.8	2.2
No. 4	2.1	2.8	1.9	2.1	1.8	2.2	1.4	1.8	1.6	2.0	2.2	1.5
No. 5	1.8	2.2	1.7	2.1	1.3	2.0	2.0	2.5	1.6	2.1	1.8	1.7
No 6	1.7	2.6	1.7	1.4	1.4	1.6	1.4	2.4	1.6	1.0	1.6	2.3
Average	2.4	2.7	2.4	2.0	1.8	2.1	1.9	2.3	1.8	1.9	1.9	2.1

Cement	7 Day						28 Days					
	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
Percentage Mean Variations of Compressive Strength between Laboratories (Values from Table III).												
No. 1	3.7	5.6	7.1	8.9	7.5	9.4	4.2	6.9	3.6	6.7	4.5	2.3
No. 2	1.3	3.8	1.7	9.7	5.6	2.3	1.5	5.2	2.9	7.0	3.9	2.5
No. 3	3.7	5.4	3.6	6.5	7.2	3.1	5.4	6.6	4.6	5.2	4.4	4.3
No. 4	2.5	4.3	3.2	4.3	4.3	3.9	3.0	4.5	2.6	3.3	1.6	2.7
No. 5	2.7	5.2	2.9	4.6	4.7	2.1	4.3	5.2	2.7	3.7	3.5	4.1
No. 6	3.0	3.9	3.0	4.4	2.9	3.6	4.1	5.2	2.3	4.6	3.3	2.0
Average	2.8	4.7	3.6	6.4	5.4	4.1	3.8	5.6	3.1	5.1	3.5	3.0
Average Percentage Mean Variations in Compressive Strength of Three Tests from Their Average (Values from Table IV).												
No. 1	4.3	2.5	2.8	3.6	4.4	3.5	2.8	1.7	2.9	2.9	2.1	3.7
No. 2	2.7	3.7	4.6	3.0	3.8	3.1	1.6	2.8	2.9	2.7	3.0	3.5
No. 3	2.2	3.4	2.6	4.2	3.6	2.4	3.1	4.1	3.0	3.3	2.5	2.7
No. 4	1.7	2.3	1.8	2.5	2 4	3.4	2.8	2.3	1.4	2.2	1.8	2.4
No. 5	1.8	4.4	3.0	1.9	2.6	3.8	2.6	2.4	1.0	2.4	2.8	2.2
No. 6	1.7	1.8	2.2	2.4	1.3	2.6	1.5	1.9	1.8	1.8	2.2	1.0
Average	2.4	3.0	2.8	2.9	3.0	3.1	2.4	2.5	2.2	2.6	2.4	2.6
Average Percentage Mean Variations in Compressive Strength of Three Test Specimens from Their Average (Values from Table V).												
No. 1	2.6	1.9	2.1	1.7	2.5	2.5	2.9	2.5	2.3	1.9	2.4	2.6
No 2	2.1	2.5	1.5	2.2	2.0	2.2	2.0	2.8	1.5	2.3	2.1	3.0
No. 3	2.1	3.3	1.9	2.3	2.1	1.9	2.0	2.3	2.1	2.3	2.2	2.6
No. 4	2.2	1.6	1.4	1.7	2.2	2.1	1.9	2.5	1.6	1.9	2.5	2.1
No. 5	1.7	3.0	1.7	1.9	2.1	2.1	2.1	2.7	1.4	2.0	2.2	1.8
No. 6	2.3	2.2	2.1	1.8	1.1	1.4	1.8	2.6	1.6	1.6	1.4	1.7
Average	2.2	2.4	1.8	1.9	2.0	2.0	2.1	2.6	1.7	2.0	2.1	2.3

TABLE VII.—RESULTS OF PHYSICAL TESTS USING A.S.T.M. METHODS C 77 – 37[5] AND C 115 – 38 T.[6]

Laboratory	Normal Consistency, per cent	Setting Time, Gillmore, hr.: min.		Soundness	Residue on No. 200 Sieve, per cent	Specific Surface, sq. cm. per g.	Tensile Strength, psi.			
		Initial	Final				1 day	3 days	7 days	28 days
				Cement No. 1						
No. 1	22.0	4:30	6:30	OK	....	1560	120	257	305	517
No. 2	22.0	4:30	8:15	OK	4.1	1658	103	235	328	447
No. 3	24.0	2:30	6:00	OK	....	1650	90	230	290	410
No. 4	23.0	3:25	5:45	OK	2.6	1535	90	217	335	452
No. 5	23.2	4:30	9:45	....	....	....	90	225	305	475
No 6	23.0	5:05	9:15	OK	....	1635	95	255	305	450
Average	23.0	4:05	7:35		3.4	1608	98	237	311	459
				Cement No. 2						
No. 1	24.0	3:15	5:15	OK	....	1760	138	317	418	525
No. 2	24.5	3:35	6:50	OK	2.0	1858	152	310	370	455
No. 3	24.0	3:00	4:40	OK	....	1875	170	305	350	445
No. 4	24.0	3:45	5:50	OK	0.7	1785	130	277	393	490
No. 5	24.0	3:45	7:30	....	....	....	140	285	370	485
No. 6	24.0	4:40	8:10	OK	....	1855	140	300	390	455
Average	24.0	3:40	6:22		1.4	1827	145	299	382	476
				Cement No. 3						
No. 1	24.0	3:15	5:15	OK	....	1630	147	278	372	465
No. 2	25.0	3:15	5:45	OK	4.2	1758	150	278	340	442
No. 3	24.0	2:10	4:30	OK	....	1805	130	270	335	420
No. 4	23.5	3:30	6:00	OK	4.0	1610	140	258	350	437
No. 5	24.0	4:00	7:30	....	....	....	135	275	350	465
No. 6	25.0	3:35	6:10	OK	....	1810	115	275	375	420
Average	24.5	3:17	5:52		4.1	1732	136	272	354	442
				Cement No. 4						
No. 1	27.0[a]	4:00	5:45	OK	....	2590	322	448	515	548
No. 2	28.0	4:15	6:55	OK	0.2	2558	325	453	470	513
No. 3	28.0	2:50	4:20	OK	....	2555	245	425	475	535
No. 4	26.0	2:30	4:35	OK	0.0	2465	323	418	452	537
No. 5	28.0	4:15	8:00	....	....	....	305	435	485	555
No. 6	26.5	4:30	7:00	OK	....	2435	330	475	535	520
Average	27.5	3:43	6:06		0.1	2521	308	442	489	535
				Cement No. 5						
No. 1	28.0	2:45	4:30	OK	....	2480	240	390	507	550
No 2	27.5	3:00	5:45	OK	0.2	2583	265	430	453	502
No. 3	27.5	2:40	4:00	OK	....	2440	225	370	440	545
No. 4	26.0	3:45	6:00	OK	0.0	2525	282	367	468	563
No. 5	28.0	4:30	7:00	....	....	....	240	400	500	555
No. 6	29.0	3:30	6:00	OK	....	2500	255	405	510	535
Average	27.5	3:22	5:32		0.1	2506	251	394	480	542
				Cement No. 6						
No. 1	29.5	2:00	3:30	OK	....	2970	313	430	445	540
No. 2	30.0	2:30	4:50	OK	0.2	3085	330	430	447	505
No. 3	28.0	1:35	2:50	OK	....	2820	305	395	450	495
No. 4	....	....	....	....	....	....	....	....	....	....
No. 5	30.0	3:15	5:45	....	....	....	265	410	465	490
No. 6	29.0	2:30	4:40	OK	....	2900	290	415	465	495
Average	29.5	2:22	4:19		0.2	2944	301	416	454	505

[a] Not determined exactly due to hesitation set.

TABLE VIII.—RESULTS OF CHEMICAL ANALYSES USING A.S.T.M. METHODS C 114 - 39.[4]

Laboratory	Silicon Dioxide, SiO_2, per cent	Aluminum Oxide, Al_2O_3, per cent	Ferric Oxide, Fe_2O_3, per cent	Calcium Oxide, CaO, per cent	Magnesium Oxide, MgO, per cent	Sulfur Trioxide, SO_3, per cent	Loss on ignition, per cent	Insoluble Residue, per cent
CEMENT NO. 1								
No. 1	....	....	....	....	....	....	....	....
No. 2	24.00	4.27	3.45	64.50	1.30	1.39	0.66	....
No. 3	24.10	4.28	3.77	63.55	1.16	1.32	1.28	....
No. 4	23.76	4.66	3.54	64.48	1.35	1.36	0.74	....
No. 5	....	....	....	....	....	....	....	....
No. 6	23.99	4.53	3.56	64.17	1.37	1.34	0.67	0.05
Average	23.96	4.44	3.58	64.18	1.30	1.35	0.84	0.05
CEMENT NO. 2								
No. 1	....	....	....	....	....	....	....	....
No. 2	21.88	5.61	2.13	63.58	3.85	1.63	0.82	....
No. 3	22.30	5.23	2.43	63.20	3.48	1.54	1.21	....
No. 4	21.74	6.30	1.82	63.42	3.80	1.60	0.97	....
No. 5	....	....	....	....	....	....	....	....
No. 6	21.82	5.87	2.17	63.00	3.83	1.62	1.26	0.07
Average	21.94	5.75	2.14	63.30	3.74	1.60	1.07	0.07
CEMENT NO. 3								
No. 1	....	....	....	....	....	....	....	....
No. 2	21.54	6.29	2.09	64.99	2.01	1.70	0.75	....
No. 3	21.40	6.39	2.36	65.00	1.86	1.60	1.30	....
No. 4	21.20	6.63	2.07	65.18	2.09	1.70	1.02	....
No. 5	....	....	....	....	....	....	....	....
No. 6	21.41	6.55	2.11	65.05	2.19	1.71	0.86	0.11
Average	21.39	6.47	2.16	65.06	2.04	1.68	0.98	0.11
CEMENT NO. 4								
No. 1	....	....	....	....	....	....	....	....
No. 2	20.84	4.68	3.60	65.84	1.22	2.27	1.08	....
No. 3	21.15	4.47	3.93	65.15	1.09	2.06	1.94	....
No. 4	20.64	4.97	3.63	65.72	1.19	2.28	1.33	....
No. 5	....	....	....	....	....	....	....	....
No. 6	20.74	4.81	3.66	65.31	1.22	2.30	1.19	0.17
Average	20.84	4.73	3.71	65.51	1.18	2.23	1.39	0.17
CEMENT NO. 5								
No. 1	....	....	....	....	....	....	....	....
No. 2	21.12	5.37	1.95	65.17	2.85	2.09	1.10	....
No. 3	21.05	5.32	2.28	65.05	2.63	1.97	1.58	....
No. 4	20.67	5.71	1.95	65.46	2.79	2.05	1.20	....
No. 5	....	....	....	....	....	....	....	....
No. 6	20.88	5.58	1.99	64.91	2.89	2.05	1.13	0.07
Average	20.93	5.50	2.04	65.15	2.79	2.04	1.25	0.07
CEMENT NO. 6								
No. 1	....	....	....	....	....	....	....	....
No. 2	22.05	4.55	2.05	63.97	3.02	2.20	1.71	....
No. 3	22.00	4.61	2.59	63.65	2.72	2.09	2.16	....
No. 4	....	....	....	....	....	....	....	....
No. 5	....	....	....	....	....	....	....	....
No. 6	21.97	4.81	2.11	63.60	3.01	2.16	1.52	0.13
Average	22.01	4.66	2.25	63.74	2.92	2.15	1.80	0.13

standing in one or more respects. It is evident that the mortars did not exhibit the same characteristics in the different laboratories, in the case of each cement. In general, water gain occurred with cements 1, 2, and 3 in all mixes, but in fewer instances with cements 4, 5, and 6. However, one laboratory reported no water gain on any mix except B-2 and with cements 1 and 2 only. The mortars B-1 and B-1L were considered difficult to mix and mold by at least two laboratories. The B-2 mortar was rated too wet by some operators with cements 1, 2, and 3, and too sticky with cements 3, 4, and 6. One laboratory rated B-4 as the best all-around mortar with B-3 second, while another rated these two mortars "not much improvement over C 109." Another laboratory rated B-3 and B-4 mortars fair as to workability and placeability, and reported no water gain with any of the cements. A study of the comments leads to the conclusion that the C 109 mortar is as satisfactory, if not more so, for testing purposes as are the other mortars from the standpoint of the several characteristics as a whole.

In Table II are given the average flow results and the average changes in the basic mixes. The increments of cement only are given since the specified cement-water ratio of 1.88 by weight was maintained in every case.

In Table III are given the average compressive strength results obtained by each laboratory on each cement.

Attention is called to the wide difference in flow results obtained with mortars B-2 and C 109 where the quantities of cement, sand, and water were held constant. The strength results were not affected by these apparent differences in consistency, and the uniformity was practically as good as that of the other mixes where the flows were held constant (75 to 90 per cent) by changing the ratio of cement-water paste to sand. It should be noted that in keeping the flows within the 75 to 90 per cent range some of the laboratories had to change the basic mix, either leaner or richer, over quite a range with the same cements. The flow results of any given laboratory are generally consistent with all of the cements and all of the mortars; that is, a laboratory obtained high, medium or low results consistently.

In Table III the percentage mean variations between laboratories are given also, and in Tables IV and V similar data for the three rounds of tests and for the three test specimens for a period. These percentage mean variations are summarized in Table VI.

There is not much difference in the uniformity of strength results of the various mortars as judged by the average percentage mean variation. Mortars B-1 and B-2 have the lowest figures. The grand average percentage mean variations, all test periods, are as follows:

	B-1 Mix	B-1L Mix	B-2 Mix	B-3 Mix	B-4 Mix	C 109 Mix
Between laboratories	4.6	5.6	5.1	7.0	6.4	5.8
Between rounds in same laboratory	3.3	3.5	3.2	3.2	3.3	3.7
Between cubes at same periods	2.1	2.5	1.9	1.9	1.9	2.1
Average	3.3	3.9	3.4	4.0	3.9	3.9

In Table VII are given other physical test results, and in Table VIII the chemical analyses of the six cements used.

CONCLUSIONS

1. Based upon the comments of the laboratory operators as to workability, ease of mixing, moldability, and water retention, the C 109 mortar appears to be as satisfactory, if not more so, for testing purposes as the other mortars investigated.

2. The B-2 (pebble) mortar, having a wetter consistency, higher water-cement

ratio, and fixed porportions of cement, sand, and water, was somewhat easier to mix and mold than the other pebble mortars (B-1 and B-1L), produced good uniformity of strength results, but was generally rated too wet and too sticky.

3. The local pebbles were practically as satisfactory as the low-absorptive pebbles.

4. When the average compressive strength results of the C 109 mortar are charted against those of the B-1, B-1L, B-3, and B-4 mortars the curves are nearly straight lines and are practically the same as those shown in Fig. 1 of the 1938 report,[1] and when charted against the B-2 mortar a straight-line relation is indicated.

FINAL SUMMARY OF COOPERATIVE INVESTIGATION OF SEVERAL MORTARS COMPARED WITH DATA FURNISHED BY NATIONAL BUREAU OF STANDARDS

The data in Table IX submitted by the National Bureau of Standards consist of compressive strength results of the six cements, using 1:4 standard Ottawa sand and 10 per cent mixing water. The mortar was placed in the cube molds by means of 1-min. vibration with a rotary vibrator having an amplitude of 0.007 in. and rotating at the rate of 83 cycles per second. These results are more uniform than plastic mortar results and appear to have a very good relation to the plastic mortar strength of the cements. It so happens that the strengths obtained by this method are practically the same as the average strengths obtained with the C 109 mortar.[2]

The summary in Table X shows the average strength results and the percentage mean variations obtained by the National Bureau of Standards (N.B.S.), using vibrated mortar, and the average strength results and the percentage mean variations obtained by the six laboratories using the C 109 mortar.[2] It should be noted that the results from the Bureau are more uniform than the average results from the six laboratories using the plastic mortar in spite of the fact that the uniformity of results from the latter is improved by the averaging process.

The vibration method appears to have excellent possibilities and should be investigated further.

The Working Committee recognizes that the present Tentative Method C 109 – 37 T[2] has shortcomings, several of which, however, are of a minor nature. The principal difficulty is that it does not take care of variations in consistency due to differences in cements, in laboratory conditions, and in grading of the testing sand. Occasionally a batch of mortar is too dry or too wet for a satisfactory molding consistency, and if too dry either more water must be added or a greater tamping pressure than that specified must be applied in order to make satisfactory cubes. Theoretically, it seems reasonable to add water and thereby penalize the strength of the harsh-working cement, but in practice the variations in consistency between laboratories due to laboratory conditions are of far greater magnitude than those due to differences in portland cements or to differences in grading of the testing sand, as demonstrated by the present series of tests and by the previous series. For this reason the fixed cement-water ratio together with the fixed cement-sand ratio should be retained until a satisfactory method for measuring consistency has been found or until some way has been devised to get around this obstacle. The dry-vibrated mortar suggested by the National Bureau of Standards not only appears to offer improved uniformity but may also prove to be a satisfactory method for eliminating the consistency problem.

It is suggested that a study of testing machine data may disclose information which, if followed, would result in improvement in uniformity of strength results, and the committee has arranged to procure the data on testing machines obtained on the Cement Reference Laboratory comparative samples.

TABLE IX.—RESULTS OF ADDITIONAL COMPRESSIVE STRENGTH TESTS MADE BY THE NATIONAL BUREAU OF STANDARDS LABORATORY IN CONNECTION WITH INVESTIGATION OF MORTARS BY SEVEN LABORATORIES, SPONSORED BY WORKING COMMITTEE ON PLASTIC MORTAR TESTS.

The plastic mortar cubes of each cement were made with 1:4 standard Ottawa sand by weight, and 10 per cent mixing water. The mortar was placed in cube molds and vibrated 1 min. with rotary vibrator, 0.007 in. amplitude and 83 cycles per second.

Date of Test	Compressive Strength of 2-in. Cubes, psi.											
	Cement No. 1		Cement No. 2		Cement No. 3		Cement No. 4		Cement No. 5		Cement No. 6	
	3 days	7 days	3 days	7 days	3 days	7 days	3 days	7 days	3 days	7 days	3 days	7 days
July 19, 1939	1065 1060 1130	1660 1700 1635	1565 1630 1615	2820 2985 3005	1485 1495 1540	2575 2580 2670	3260 3260 3225	4795 4735 4780	2890 2850 2885	5075 5030 5070	3550 3465 3570	4785 4900 4840
Average	1085	1665	1605	2935	1505	2610	3250	4770	2875	5060	3530	4840
Percentage mean variation	2.8	1.4	1.6	2.7	1.5	1.6	0.5	0.5	0.6	0.4	1.2	0.8
July 21, 1939	1070 1085 1040	1655 1670 1655	1545 1575 1585	3060 3050 3120	1495 1470 1510	2765 2840 2785	3185 3225 3220	4755 4880 4950	2975 2925 2895	5130 5265 5260	3525 3490 3460	4845 5015 5085
Average	1065	1660	1570	3075	1490	2795	3210	4860	2930	5220	3490	4980
Percentage mean variation	1.6	0.4	1.0	0.9	1.0	1.0	0.5	1.5	1.0	1.1	0.9	1.8
July 24, 1939	1110 1115 1065	1630 1640 1685	1640 1620 1655	3045 2965 3030	1555 1555 1505	2600 2640 2625	3285 3305 3340	5040 4955 5100	2925 2965 2965	5465 5315 5285	3485 3715 3620	4955 5015 5090
Average	1095	1650	1640	3015	1540	2620	3310	5030	2950	5355	360	5020
Percentage mean variation	2.0	1.3	0.7	1.1	1.4	0.6	0.6	1.0	0.6	1.4	2.3	0.9
Grand average, psi	1080	1660	1605	3010	1510	2675	3255	4885	2920	5210	3540	4945
Average percentage mean variation	2.1	1.0	1.1	1.6	1.3	1.1	0.5	1.0	0.7	1.0	1.5	1.2
Percentage mean variation between rounds	1.1	0.3	1.4	1.6	1.2	3.0	1.1	1.9	1.0	2.0	1.2	1.5

TABLE X.—COMPARISON OF DATA FROM NATIONAL BUREAU OF STANDARDS TESTS WITH RESULTS FROM SIX COOPERATING LABORATORIES ON MORTAR C 109.

	Cement No. 1		Cement No. 2		Cement No. 3		Cement No. 4		Cement No. 5		Cement No. 6	
	3 days	7 days	3 days	7 days	3 days	7 days	3 days	7 days	3 days	7 days	3 days	7 days
N.B.S.[a]	1080	1660	1605	3010	1510	2675	3255	4885	2920	5210	3540	4945
C 109[a]	995	1644	1539	3069	1382	2481	3305	4655	2977	5201	3459	4896
N.B.S.[b]	2.1	1.0	1.1	1.6	1.3	1.1	0.5	1.0	0.7	1.0	1.5	1.2
C 109[b]	2.3	2.5	2.6	2.2	2.2	1.9	1.5	2.1	1.7	2.1	2.3	1.4
N.B.S.[c]	1.1	0.3	1.4	1.6	1.2	3.0	1.1	1.9	1.0	2.0	1.2	1.5
C 109[c]	3.1	3.5	4.0	3.1	3.0	2.4	3.3	3.4	1.9	3.8	2.1	2.6

[a] Average compressive strength in pounds per square inch, results from Tables IX and III.
[b] Percentage mean variation between individual test specimens at a period, results from Tables IX and V.
[c] Percentage mean variation between three rounds of tests, results from Tables IX and IV.

The Working Committee believes that none of the plastic mortars covered in the investigation shows sufficient superiority over the C 109 mortar to warrant a change in type of mortar and it recommends that the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 37 T) be retained as tentative for another year in order that the committee may investigate suggestions for possible improvement resulting from a study of the effect of different types, capacities, etc., of testing machines, and in order to investigate the high-frequency vibration method for molding cubes suggested by the National Bureau of Standards.

Respectfully submitted on behalf of the Working Committee,

O. L. MOORE,
Chairman.

REPORT ON COOPERATIVE STUDIES OF VARIOUS TYPES OF SOUNDNESS TESTS

Sponsored by Working Committee on Volume Change and Soundness

In March, 1939, arrangements were made with several of the members of Committee C-1, and others interested, to participate in a program of soundness tests sponsored by the Working Committee on Volume Change and Soundness of Portland Cement. During the past year the Working Committee has received reports from the cooperating laboratories and has made a study of the results.

The principal aims of this work have been to determine, through a cooperative series of tests, the effect of certain variations in the proposed method of conducting the autoclave test, to explore the possibility of using a modified pat as a test specimen in the autoclave test, and to study the autoclave expansions of neat bars in relation to the strengths of corresponding autoclaved and unautoclaved neat and standard mortar briquets.

This report gives the results of this cooperative series in which 30 laboratories participated by making tests on several cements which the various laboratories had available for routine tests.

Cooperating Laboratories: The 30 laboratories that cooperated in making the tests in whole or in part are listed below:

Allentown Portland Cement Co., Catasauqua, Pa., M. D. Olver, Supervising Chemist.

Alpha Portland Cement Co., Easton, Pa., L. Anderson Jr., Chemical Engineer.

Bessemer Limestone and Cement Co., Youngstown, Ohio, R. E. Roscoe, Vice-President.

California Portland Cement Co., Colton, Calif., W. C. Hanna, Chief Chemist and Chemical Engineer.

State of California Dept. of Public Works, Division of Highways, Materials and Research Dept., Sacramento, Calif., T. E. Stanton, Materials and Research Engineer.

Canada Cement Co., Ltd. (four plant laboratories), Montreal, Quebec, Canada, A. G. Fleming, Chief Chemist.

Federal Portland Cement Co., Inc., Buffalo, N. Y., J. F. Barton, Chief Chemist.

Froehling and Robertson, Inc., Richmond, Va., G. J. Durant, President.

Glens Falls Portland Cement Co., Glens Falls, N. Y., C. T. Raber, Chief Chemist.

Green Bag Cement Co. of Pennsylvania, Pittsburgh, Pa., L. N. Bryant, Research Engineer.

State Highway Comm. of Kansas, Manhattan, Kans., W. E. Gibson, Engineer of Tests.

Keystone Portland Cement Co., Bath, Pa., H. H. Leh, Gen. Operating Manager.

Kosmos Portland Cement Co., Louisville, Ky., E. E. Berger, Chief Chemist.

Lawrence Portland Cement Co., Northampton, Pa., L. A. Eisenhard, Chief Chemist.

Lehigh Portland Cement Co., Allentown, Pa., R. N. Young, Chemical Engineer.

Lone Star Cement Corp. (four plant laboratories), New York, N. Y., H. R. Durbin, Chief Chemist.

Medusa Portland Cement Co., Wampum, Pa., H. Vanderwerp, Vice-President.

Pennsylvania-Dixie Cement Corp., Nazareth, Pa., W. H. Klein, Vice-President.

Portland Cement Association, Chicago, Ill., H. F. Gonnerman, Manager of Laboratory.

Southwestern Portland Cement Co., Osborn, Ohio, G. E. Warren, Vice-President and Manager.

Thompson and Lichtner Co., Inc., Boston, Mass., M. N. Clair, Vice-President.

Universal Atlas Cement Co., New York,

N. Y., O. L. Moore, Director, Tests and Research.
U. S. Dept. of the Interior, Bureau of Reclamation, Denver, Colo., R. F. Walter, Chief Engineer.
Whitehall Cement Mfg. Co., Cementon, Pa., M. H. Meighan, Chief Chemist.

Scope of Tests.[1]—The principal items under investigation in these tests were:

1. Effect of age on autoclave expansion of neat cement bars cured moist for 1 day and 14 days before test (groups 1 and 1A of Table I).
2. Effect of molding procedure on autoclave expansion of neat cement bars (groups 1 and 2 of Table I).
3. Comparison of autoclave test of neat bars and modified neat pats at 1 day with the standard A.S.T.M. soundness test of neat pats (groups 3 and 4 of Table I).
4. Strength and volume change of moist-cured and autoclaved neat cement briquets (groups 5 and 6 of Table I).
5. Strength and volume change of moist-cured and autoclaved 1:3 standard mortar briquets (groups 7 and 8 of Table I).
6. Volume change of neat cement cylinders in the "LeChatelier" test (groups 9 and 9A of Table I).

Cements.—The cements used in these tests comprised a variety of both commercial and experimental cements. Each laboratory obtained the cements on its own initiative and so far as is known no two laboratories tested the same cements.

DATA AND DISCUSSION OF TESTS

Results of tests reported by the cooperating laboratories are presented in Table I. Details of the procedure followed in the tests are outlined in the notes at the head of the table. In this table the cements for each laboratory are arranged according to type, and the cements of a given type are arranged in the order of their expansions in the autoclave as determined at the usual 1-day period.

Effect of Age on Autoclave Expansion.—Of the 221 cements tested using both 1-day and 14-day moist-cured bars, 206 or 94 per cent of the cements showed approximately the same or less expansion when tested at 14 days than when tested at 1 day. The remaining 15 cements, which had appreciably higher autoclave expansions at 14 days than at 1 day, are listed in Table II.

The 15 cements in Table II are listed in the order of their autoclave expansions at 1 day, the first 4 cements having expansions less than 0.5 per cent, the next 2 having expansions between 0.5 and 1 per cent, and the remaining 10 cements having expansions in excess of 1 per cent. Of the 6 cements having autoclave expansions of less than 1 per cent at 1 day, 4 showed much higher expansions at 14 days than at 1 day. This was also the case for several of the cements having expansions at 1 day over 1 per cent. The other cements listed show relatively small differences between the expansions at 1 day and 14 days, and such differences as were reported may possibly be explained by differences in conducting the tests.

The higher expansions reported in the 14-day tests for some of these 15 cements are contrary to experience, since aging of the specimen has been found to reduce the expansion. Attention is called to the results for laboratory No. 24 listed in Table II which show that repeat tests in the case of cement marked 2 did not confirm the high expansion found at the later age in the earlier test. The reverse is true in the case of cement

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[1] See "Outline of Cooperative Studies of Various Types of Soundness Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 312 (1939).

TABLE I.—COOPERATIVE STUDIES OF VARIOUS TYPES OF SOUNDNESS TESTS.

Preparation, Curing, and Testing of Specimens.

For each cement, all neat-cement specimens (groups 1 to 6 and 1A) except those for the LeChatelier test (group 9) were cast from a single batch composed, in general, of 2500 g. of cement and sufficient water for normal consistency. 1:3 standard sand mortar briquets (groups 7 and 8) were cast from a single batch composed of 250 g. of cement, 750 g. of standard sand, and sufficient water for normal consistency.

Group 1.—Neat-cement specimens either 1 by 1 by 11¼-in. or 1 by 1 by 6¼-in. molded in three layers, were cured 24 hr. in molds in a moist closet, and tested in accordance with the Proposed Method of Test for Autoclave Expansion of Portland Cement.[2]

Group 1A.—Same as group 1 except specimens were removed from the molds and cured for 13 additional days in a moist closet or fog room before being subjected to autoclave test.

Group 2.—Same as group 1 except specimens were molded in either one or two layers, each layer being compacted sufficiently with thumbs or forefingers to obtain a uniform specimen.

Group 3.—Modified pat specimens prepared in accordance with the Standard Methods of Sampling and Physical Testing of Portland Cement (A.S.T.M. Designation: C 77 - 37),[3] except that beveled edge of pat was cut off with a circular cooky cutter forming a pat 2⅝-in. in diameter having a thickened edge. After 24 hr. in the moist closet, the pat was subjected to the same autoclave test as in group 1. Visual observations were made for unsoundness as manifested by change in volume causing distortion, cracking, checking, swelling, or disintegration. Pats were examined both when wet and dry, with a reading glass, unless otherwise noted.

Group 4.—Pats were made, cured and tested in accordance with the A.S.T.M. Standard Methods C 77 - 37.[3]

Groups 5 and 7.—Briquets were molded in accordance with the A.S.T.M. Standard Methods C 77 - 37,[3] cured 24 hr. in moist closet, removed from molds and cured 6 hr. in moist closet or fog room, and then tested for tensile strength at age of 30 hr.

Groups 6 and 8.—Briquets were molded in same manner as in groups 5 and 7. After 24 hr. in moist closet briquets were removed from molds and subjected to same autoclave test as in group 1. Tested for tensile strength at age of 30 hr.

Group 9.—The 30 by 30-mm. neat-cement cylinders were made, cured and tested in accordance with usual methods. The period of storage in boiling water varied from 3 to 5 hr. For details of the method used, reference may be made to: "Portland Cement," by R. K. Meade, p. 620 (1926); "The Chemistry of Cement and Concrete," by F. M. Lea and C. H. Desch, p. 213 (1935); and also The Standard Specifications of the British Standards Institution.

Group 9A.—Same as group 9 except specimens were subjected to same autoclave test as in group 1.

Number of Specimens.—Values and observations for 1 by 1 by 11¼-in. or 1 by 1 by 6¼-in. neat-cement bars and neat-cement pats (groups 1 to 4) represent one test only. Strength values and dimensions of briquets (groups 5 to 8) are the average of tests on three briquets.

Type of Cement	Reference Mark	A.S.T.M. Normal Consistency, per cent	Autoclave Tests of 1 by 1 by 11¼-in. Neat-Cement Bars					Modified Autoclave Soundness Pat	A.S.T.M. Standard Soundness Pat	Neat-Cement Briquets					1:3 Standard Sand Mortar Briquets					LeChatelier Expansion Test	
			Proposed 1938 Method[2]		Modified Molding Procedure[4]					30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	In Boiling Water	5-hr. Autoclave Test
			Expansion, per cent, Bars Cured Moist for		Number of Layers	Total Number of Tamps	Expansion, per cent, Bars Cured Moist for 1 day				Expansion at Waist Line, in.		Tensile Strength, psi.			Expansion at Waist Line, in.		Tensile Strength, psi.			
			1 day	14 days							Width	Thickness				Width	Thickness				
Group No.			1	1A	2	2	2	3	4	5	6	6	6		7	8	8	8		9	9A
LABORATORY NO. 1																					
Special	A	....	0.16	0.07	....	....	0.16	OK	OK	347	0.003	0.001	430	1.24	203	0.001	0.002	170	0.84	....	....
	B	....	0.26	0.11	....	....	0.26	OK	OK	495	0.002	0.003	437	0.88	200	0.002	0.002	190	0.95	....	....
	E	....	0.99	0.24	....	....	0.84	OK	OK	350	0.004	0.008	337	0.96	180	0.002	0.004	177	0.98	....	....
	F	....	1.11	0.27	....	....	1.06	OK	OK	452	0.006	0.011	265	0.59	177	0.001	0.002	187	1.06	....	....
	C	....	2.10	8.94	....	....	2.96	OK	OK	507	0.022	0.019	303	0.60	178	0.009	0.011	155	0.87	....	....
	D	....	2.51	3.57	....	....	3.33	OK	OK	435	....	....	363	0.83	162	....	....	158	0.97	....	....
LABORATORY NO. 2																					
Normal	418	....	−0.02	−0.04	....	....	−0.02	OK	OK	427	....	....	510	1.19	145	....	....	224	1.54	....	....
	416	....	0.39	0.16	....	....	0.39	[a]	OK	435	....	....	385	0.88	162	....	....	182	1.12	....	....
	415	....	0.61	0.18	....	....	0.62	[b]	OK	490	....	....	342	0.69	182	....	....	210	1.15	....	....
	414	....	1.20	0.41	....	....	1.24	[c]	OK	342	....	....	482	1.29	217	....	....	187	0.86	....	....
Low Heat	417	....	0.18	0.09	....	....	0.18	[a]	OK	420	....	....	403	0.96	137	....	....	192	1.40	....	....

LABORATORY NO. 3

Normal	4	....	0.12	0.03	....	....	0.13	OK	OK	513	....	....	490	0.96	247	....	....	242	1.00	....	....
	9	....	0.12	0.04	....	....	0.13	OK	OK	530	....	....	512	0.97	262	....	....	272	1.04	....	....
	8	....	0.13	0.05	....	....	0.13	OK	OK	460	....	....	460	1.00	245	....	....	237	0.97	....	....
	10	....	0.14	0.04	....	....	0.13	OK	OK	507	....	....	533	1.05	242	....	....	250	1.03	....	....
	3	....	0.14	0.06	....	....	0.14	OK	OK	462	....	....	445	0.96	237	....	....	257	1.08	....	....
	5	....	0.14	0.04	....	....	0.14	OK	OK	473	....	....	485	1.03	250	....	....	260	1.04	....	....
	1	....	0.14	0.03	....	....	0.14	OK	OK	522	....	....	478	0.92	223	....	....	220	0.99	....	....
	6	....	0.14	0.03	....	....	0.14	OK	OK	443	....	....	448	1.00	243	....	....	233	0.96	....	....
	7	....	0.15	0.07	....	....	0.15	OK	OK	483	....	....	470	0.97	250	....	....	262	1.05	....	....
	2	....	0.16	0.08	....	....	0.16	OK	OK	455	....	....	553	1.21	215	....	....	253	1.18	....	....

LABORATORY NO. 4

Lab.	2801	....	0.89	....	....	....	....	OK	OK	....	0	0	228	....	....	....	....	....	....	....	....
	2786	....	0.98	....	....	....	....	OK	OK	....	0	0	286	....	....	....	....	....	....	....	....
	2787	....	1.04	....	....	....	....	....	OK	....	1/64	1/64	745	....	....	....	....	....	....	....	....
	2869	....	1.08	....	....	....	....	OK	OK	289	1/64	1/64	333	1.15	....	....	....	....	....	....	....
	2821	....	1.13	....	....	....	....	....	OK	....	0	0	210	....	....	....	....	....	....	....	....
	2871	....	1.24	....	....	....	....	d	OK	346	2/64	2/64	236	0.68	....	....	....	....	....	....	....
	2788	....	1.91	....	....	....	....	....	OK	....	1/64	2/64	182	....	....	....	....	....	....	....	....
	2822	....	2.17	....	....	....	....	OK	OK	98	1/64	5/64	190	1.94	....	....	....	....	....	....	....
	2798	....	2.40	....	....	....	....	....	OK	....	1/64	2/64	46	....	....	....	....	....	....	....	....
	2831	....	4.15	....	....	....	....	OK	OK	170	1/64	1/64	388	2.28	....	....	....	....	....	....	....
	2870	....	5.00	....	....	....	....	e	OK	343	8/64	9/64	too big	....	....	....	....	....	....	....	....
	2866	....	8.65	....	....	....	....	e	OK	290	7/64	4/64	43	0.15	....	....	....	....	....	....	....
Normal	2876	....	0.17	0.16	....	....	....	OK	OK	504	0	0	610	1.21	....	....	....	....	....	....	....
	2878	....	0.18	0.05	....	....	....	OK	OK	449	0	1/64	403	0.90	....	....	....	....	....	....	....
	2877	....	0.20	0.08	....	....	....	OK	OK	541	0	1/64	525	0.97	....	....	....	....	....	....	....
	2879	....	0.26	0.16	....	....	....	OK	OK	529	1/64	1/64	460	0.87	....	....	....	....	....	....	....
	2897	....	0.32	0.17	....	....	....	OK	OK	485	0	0	587	1.21	....	....	....	....	....	....	....
	2880	....	0.32	0.12	....	....	....	OK	OK	493	0	0	442	0.90	....	....	....	....	....	....	....
	2868	....	0.32	....	....	....	....	OK	OK	423	1/64	0	534	1.26	....	....	....	....	....	....	....
Plastic	2883	....	0.12	0.02	....	....	....	OK	OK	431	0	0	357	0.83	....	....	....	....	....	....	....
	2882	....	0.12	0.01	....	....	....	OK	OK	331	0	0	296	0.89	....	....	....	....	....	....	....
	2881	....	0.32	0.25	....	....	....	OK	OK	576	0	0	400	0.69	....	....	....	....	....	....	....
Pozzuolana	1492	....	0.06	0.01	....	....	....	OK	OK	288	0	0	703	2.44	....	....	....	....	....	....	....
	2884	....	0.09	−0.01	....	....	....	OK	OK	341	0	0	834	2.45	....	....	....	....	....	....	....
Hi-Early	2908	....	0.12	0.07	....	....	....	OK	OK	813	0	0	476	0.58	....	....	....	....	....	....	....
	2909	....	0.26	0.09	....	....	....	OK	OK	531	0	0	427	0.80	....	....	....	....	....	....	....
Sulfate Resisting	2912	....	0.11	0.08	....	....	....	OK	OK	419	0	0	684	1.63	....	....	....	....	....	....	....
	2913	....	0.20	0.13	....	....	....	OK	OK	498	0	0	447	0.90	....	....	....	....	....	....	....
Low-Heat	2907	....	0.13	0.01	....	....	....	OK	OK	286	0	0	575	2.01	....	....	....	....	....	....	....
U.S. Engr.	2893	....	0.01	0.11	....	....	....	OK	OK	530	0	0	582	1.10	....	....	....	....	....	....	....

TABLE I.—COOPERATIVE STUDIES OF VARIOUS TYPES OF SOUNDNESS TESTS.—*Continued*

Type of Cement	Reference Mark	A.S.T.M. Normal Consistency, per cent	Autoclave Tests of 1 by 1 by 11¼-in. Neat-Cement Bars					Modified Autoclave Soundness Pat	A.S.T.M. Standard Soundness Pat	Neat-Cement Briquets					1:3 Standard Sand Mortar Briquets					LeChatelier Expansion Test	
			Proposed 1938 Method[2]		Modified Molding Procedure[4]					30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	In Boiling Water	5-hr. Autoclave Test
			Expansion, per cent, Bars Cured Moist for		Number of Layers	Total Number of Tamps	Expansion, per cent, Bars Cured Moist for 1 day				Expansion at Waist Line, in.		Tensile Strength, psi.			Expansion at Waist Line, in.		Tensile Strength, psi.			
			1 day	14 days							Width	Thickness				Width	Thickness				
Group No.			1	1A	2	2	2	3	4	5	6	6	6		7	8	8	8		9	9A
Laboratory No. 5																					
Normal	8072	....	0.02	0.00	....	....	0.02	OK	OK	495	....	....	500	1.01	165	....	....	195	1.18	....	....
	03158	....	0.08	0.05	....	....	0.08	OK	OK	445	....	....	360	0.81	125	....	....	190	1.52	....	....
	05063	....	0.11	0.05	....	....	0.11	OK	OK	390	....	....	385	0.99	155	....	....	200	1.29	....	....
	04231	....	0.17	0.17	....	....	0.15	[f]	OK	605	....	....	395	0.65	155	....	....	175	1.13	....	....
	7348	....	0.19	2.1+	....	....	0.14	OK	OK	375	....	....	450	1.20	95	....	....	180	1.90	....	....
	2147	....	0.20	0.20	....	....	0.19	[g]	OK	420	....	....	535	1.27	145	....	....	185	1.28	....	....
	3248	....	0.22	0.20	....	....	0.22	OK	OK	515	....	....	525	1.02	195	....	....	180	0.92	....	....
	1218	....	0.22	0.20	....	....	0.14	OK	OK	460	....	....	405	0.88	175	....	....	185	1.06	....	....
Hi-Early	7349	....	0.40	9.7	....	....	0.38	OK	OK	535	....	....	435	0.81	330	....	....	185	0.56	....	....
Hi-Silica	00164	....	0.02	−0.01	....	....	0.02	OK	OK	370	....	....	825	2.23	195	....	....	425	2.18	....	....
Laboratory No. 6																					
Normal	1	....	0.51	0.28	....	....	0.54	[h]	OK	539	0	0	467	0.86	168	0	0	219	1.30	5/16 in.	....
	8	....	0.85	3.25	....	....	1.12	OK	OK	438	0	0	395	0.90	109	0	0	215	1.97	1/64 in.	31/64 in.
	4	....	1.23	0.99	....	....	1.37	OK	OK	352	0	0	388	1.10	109	0	0	230	2.11	1/16 in.	5/16 in.
	6	....	1.30	1.60	....	....	1.37	OK	OK	383	0	0	347	0.90	122	0	0	178	1.45	1/16 in.	9/32 in.
	2	....	2.70	too big	....	....	3.10	[i]	OK	338	0	0	321	0.95	104	0	0	186	1.78	13/64 in.	53/64 in.
	9	....	3.30	too big	....	....	too big	[a]	OK	392	0	0	250	0.63	107	0	0	189	1.78	1/16 in.	61/64 in.
	3	....	too big	too big	....	....	too big	OK	OK	219	0	0	192	0.87	93	0	0	227	2.44	....	....
	5	....	too big	too big	....	....	too big	[j]	OK	247	8/64	0	0	0.00	105	0	0	75	0.71	....	....
	7	....	too big	too big	....	....	too big	[j]	OK	322	4/64	0	0	0.00	136	3/64	0	60	0.44	1/8 in.	1 3/16 in.
Sulfate-Resisting	10	....	0.02	0.00	....	....	0.02	OK	OK	599	0	0	493	0.82	188	0	0	244	1.29	0	0
Laboratory No. 7																					
Normal	1	....	0.12	0.07	....	....	0.12	OK	OK	700	....	....	432	0.62	226	....	....	203	0.90	1 mm.	2 mm.
Hi-Early	2	....	0.05	0.03	1	....	0.05	OK	OK	757	....	....	692	0.91	423	....	....	320	0.76	1 mm.	1.5 mm.
Laboratory No. 8																					
Normal	(17–22)	....	0.12	....	....	....	....	....	....	530	....	....	655	1.22	....	....	....	....	....	....	....
	(9–14)	....	0.12	....	....	....	....	....	....	590	....	....	675	1.14	....	....	....	....	....	....	....
	(37–44)	....	0.14	....	....	....	....	....	....	580	....	....	608	1.05	....	....	....	....	....	....	....

LABORATORY No. 9																					
Normal	A	....	0.23	0.07	1	....	0.25	OK	OK	706	0.005	0.001	482	0.68	186	−0.001	0.003	225	1.21	....	....
LABORATORY No. 10																					
Normal	1	....	0.35	0.11	1	100	0.34	OK	OK	350	....	....	376	1.07	202	....	....	260	1.28	....	....
N. Y. 15C	2	....	0.06	0.01	2	100	0.01	OK	OK	350	....	....	485	1.38	181	....	....	257	1.42	....	....
LABORATORY No. 11																					
Normal	866	....	0.26	0.12	1	50	0.27	OK	OK	503	....	....	482	0.96	212	....	....	223	1.09	....	....
	738	....	0.40	0.23	1	50	0.42	OK	OK	438	....	....	350	0.80	135	....	....	233	1.73	....	....
	811	....	0.43	0.34	1	50	0.33	OK	OK	375	....	....	378	1.01	182	....	....	195	1.07	....	....
Hi-Early	817	....	0.17	0.09	1	50	0.18	OK	OK	400	....	....	365	0.91	340	....	....	300	0.88	....	....
Mod. Heat	804	....	0.20	0.11	1	50	0.20	OK	OK	377	....	....	527	1.40	40	....	....	200	5.00	....	....
LABORATORY No. 12																					
Normal	6	....	0.16	0.07	....	....	0.14	OK	OK	622	....	....	437	0.73	228	....	....	207	0.91	....	....
	3	....	0.63	0.29	....	....	0.58	OK	OK	488	....	....	418	0.93	265	....	....	235	0.89	....	....
	4	....	0.70	0.61	....	....	0.69	OK	OK	427	....	....	368	0.86	280	....	....	187	0.67	....	....
	2	....	0.71	....	....	....	1.64	k	OK[1]	515	....	....	447	0.87	258	....	....	217	0.84	....	....
	5	....	2.17	2.50	....	....	2.27	m	OK	485	....	....	178	0.37	262	....	....	90	0.34	....	....
Hi-Early	1	....	0.18	0.48	....	....	0.18	OK	OK[1]	467	....	....	557	1.19	343	....	....	260	0.76	....	....
LABORATORY No. 13																					
Normal	1	....	0.07	0.01	....	....	0.06	OK	OK	343	....	....	432	1.26	122	....	....	160	1.31	....	....
	5	....	0.07	0.01	....	....	0.07	OK	OK	365	....	....	375	1.03	135	....	....	215	1.60	....	....
	6	....	0.07	0.01	....	....	0.07	OK	OK	335	....	....	393	1.14	143	....	....	197	1.39	....	....
	4	....	0.08	0.00	....	....	0.08	OK	OK	343	....	....	415	1.21	122	....	....	188	1.55	....	....
	9	....	0.08	0.01	....	....	0.08	OK	OK	368	....	....	413	1.12	130	....	....	185	1.40	....	....
	2	....	0.09	0.00	....	....	0.08	OK	1 crack	338	....	....	355	1.05	117	....	....	215	1.83	....	....
	10	....	0.09	0.01	....	....	0.09	OK	OK	350	....	....	398	1.14	128	....	....	227	1.77	....	....
	3	....	0.09	0.00	....	....	0.09	OK	OK	360	....	....	405	1.13	138	....	....	230	1.66	....	....
	8	....	0.10	0.01	....	....	0.09	OK	2 cracks	342	....	....	403	1.18	135	....	....	205	1.52	....	....
	7	....	0.10	0.01	....	....	0.01	OK	OK	362	....	....	445	1.23	125	....	....	203	1.62	....	....
Portland-Pozzuolana	11	....	−0.04	−0.01	....	....	−0.04	OK	OK	475	....	....	205	0.43	172	....	....	393	2.28	....	....
	12	....	−0.03	−0.01	....	....	−0.03	OK	OK	452	....	....	250	0.55	173	....	....	380	2.20	....	....
	13	....	−0.02	0.00	....	....	−0.01	OK	OK	463	....	....	248	0.54	152	....	....	403	2.65	....	....
	14	....	−0.01	0.00	....	....	−0.01	OK	OK	493	....	....	282	0.57	170	....	....	425	2.50	....	....
Modified	17	....	0.03	0.00	....	....	0.02	OK	OK	347	....	....	388	1.12	117	....	....	257	2.20	....	....
	16	....	0.03	0.00	....	....	0.02	OK	OK	342	....	....	402	1.16	122	....	....	233	1.91	....	....
	18	....	0.03	0.01	....	....	0.03	OK	OK	362	....	....	388	1.07	127	....	....	235	1.84	....	....
	15	....	0.04	0.00	....	....	0.04	OK	OK	335	....	....	388	1.13	125	....	....	252	2.01	....	....
Waterproof	19	....	0.02	0.03	....	....	0.02	OK	OK	327	....	....	410	1.25	128	....	....	222	1.73	....	....
	20	....	0.02	0.03	....	....	0.02	OK	OK	340	....	....	398	1.16	133	....	....	225	1.70	....	....

TABLE I.—COOPERATIVE STUDIES OF VARIOUS TYPES OF SOUNDNESS TESTS.—*Continued*

Type of Cement	Reference Mark	A.S.T.M. Normal Consistency, per cent	Autoclave Tests of 1 by 1 by 11¼-in. Neat-Cement Bars					Modified Autoclave Soundness Pat	A.S.T.M. Standard Soundness Pat	Neat-Cement Briquets					1:3 Standard Sand Mortar Briquets					LeChatelier Expansion Test	
			Proposed 1938 Method[2]		Modified Molding Procedure[4]					30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	In Boiling Water	5-hr. Autoclave Test
			Expansion, per cent, Bars Cured Moist for		Number of Layers	Total Number of Tamps	Expansion, per cent, Bars Cured Moist for 1 day				Expansion at Waist Line, in.		Tensile Strength, psi.			Expansion at Waist Line, in.		Tensile Strength, psi.			
			1 day	14 days							Width	Thickness				Width	Thickness				
Group No.			1	1A	2	2	2	3	4	5	6	6	6		7	8	8	8		9	9A
										LABORATORY NO. 14											
Normal	37382	25.5	0.02	−0.02	1	100	0.02	OK	OK	485	0	0	543	1.12	210	0	0	200	0.95	....	....
	37254	23.0	0.03	−0.01	1	100	0.02	OK	OK	553	−0.001	−0.001	533	0.97	225	0.001	0	223	0.99	....	....
	37216	24.5	0.05	0.01	1	100	0.05	OK	OK	383	0	0.001	473	1.24	200	0	0.001	233	1.16	....	....
	37247	24.0	0.14	0.04	1	100	0.13	OK	OK	517	0	0	420	0.81	213	0	0.001	225	1.06	....	....
	37215	24.5	0.15	0.04	1	100	0.16	OK	OK	385	0.002	−0.001	415	1.08	183	0.001	−0.001	188	1.03	....	....
	37214	23.5	0.16	0.11	1	100	0.15	OK	OK	438	0	−0.001	638	1.46	203	0	0	207	1.02	....	....
	37245	23.5	0.17	0.05	1	100	0.17	OK	OK	503	−0.001	0	480	0.95	240	−0.001	0	210	0.88	....	....
White	37478	25.0	0.00	−0.03	1	100	0.00	OK	OK	463	−0.001	−0.001	425	0.92	183	−0.001	0.001	217	1.18	....	....
										LABORATORY NO. 15											
Normal	2	....	0.27	0.15	....	....	0.27	OK	OK	428	....	....	435	1.01	193	....	....	163	0.84	....	....
Hi-Early	5	....	0.56	0.65	....	....	0.56	OK	OK	382	....	....	373	0.98	310	....	....	193	0.62	....	....
Special	6	....	0.03	−0.01	....	....	0.03	OK	OK	370	....	....	353	0.95	318	....	....	323	1.01	....	....
	4	....	0.04	0.01	....	....	0.04	OK	OK	472	....	....	547	1.16	207	....	....	197	0.95	....	....
	1	....	0.91	0.34	....	....	0.87	OK	OK	402	....	....	355	0.88	193	....	....	160	0.83	....	....
	3	....	1.42	0.83	....	....	1.40	OK	OK	413	....	....	372	0.90	188	....	....	168	0.89	....	....
										LABORATORY NO. 16											
Laboratory Ground	1	....	0.18	....	....	....	....	....	....	492	....	....	613	1.24	275	....	....	329	1.20	....	....
	2	....	0.28	....	....	....	....	....	....	452	....	....	625	1.38	348	....	....	400	1.15	....	....
	5	....	0.28	....	....	....	....	....	....	560	....	....	665	1.19	295	....	....	327	1.11	....	....
	3	....	0.40	....	....	....	....	....	....	480	....	....	652	1.36	275	....	....	302	1.02	....	....
	6	....	0.81	....	....	....	....	....	....	577	....	....	435	0.75	282	....	....	310	1.10	....	....
	4	....	10.0+	....	....	....	....	....	....	438	....	....	too large for clips, soft		298	....	....	too large for clips		....	....

LABORATORY No. 17																					
Normal	4	....	0.37	....	....	....	....	OK	OK	397	0.004	0.004	455	1.15	205	0	0.001	207	1.01	....	....
	1	....	0.39	....	....	....	....	OK	OK	437	0.003	0.002	477	1.09	197	0.002	0.001	197	1.00	....	....
	5	....	0.41	....	....	....	....	OK	OK	430	0.005	0.005	458	1.07	163	0.001	0.001	205	1.26	....	....
	3	....	0.42	0.18	....	....	....	OK	OK	468	0.004	0.006	425	0.91	185	0.001	0.001	208	1.12	....	....
	2	....	0.42	....	....	....	....	OK	OK	383	0.005	0.006	478	1.25	185	0.001	0.001	210	1.14	....	....
	6	....	0.56	0.24	....	....	....	OK	OK	402	0.006	0.007	445	1.11	147	0.002	0.001	200	1.36	....	....
Hi-Early	11	....	0.08	0.05	....	....	....	OK	OK	417	0	0.001	533	1.28	330	....	....	322	0.98	....	....
	12	....	0.09	0.04	....	....	....	OK	OK	498	0	0	487	0.98	347	....	....	347	1.00	....	....
Dark, Normal	10	....	0.04	0.04	....	....	....	OK	OK	442	0	0	533	1.21	258	....	....	312	1.21	....	....
	7	....	0.10	....	....	....	....	OK	OK	440	0.001	0.001	437	0.99	223	0.001	0	223	1.00	....	....
N. J. Specifications	8	....	0.04	....	....	....	....	OK	OK	438	0	0.001	525	1.20	213	0	....	282	1.32	....	....
N. Y. 15C	9	....	0.03	0.01	....	....	....	OK	OK	393	0	0	562	1.43	272	....	....	338	1.24	....	....
LABORATORY No. 18																					
Normal	8	....	0.35	0.14	2	50	0.34	n	OK	472	....	....	345	0.73	200	....	....	188	0.94	....	....
	7	....	0.41	0.19	2	50	0.46	OK	OK	437	....	....	367	0.84	160	....	....	195	1.22	....	....
	10	....	0.46	0.20	2	50	0.50	i	i	512	....	....	380	0.74	205	....	....	180	0.88	....	....
	4	....	0.49	0.30	2	50	0.48	OK	OK	537	....	....	403	0.75	153	....	....	170	1.11	....	....
	11	....	0.49	0.20	2	50	0.55	i	i	588	....	....	343	0.58	190	....	....	200	1.05	....	....
	1	....	0.50	0.18	2	50	0.51	OK	OK	480	....	....	483	1.01	183	....	....	188	1.03	....	....
	2	....	0.70	0.38	2	50	0.70	n	OK	453	....	....	442	0.98	210	....	....	175	0.83	....	....
	12	....	0.73	0.38	2	50	0.72	OK	OK	478	....	....	310	0.65	187	....	....	182	0.97	....	....
	6	....	0.95	0.38	2	50	1.29	OK	OK	408	....	....	295	0.72	135	....	....	183	1.36	....	....
	9	....	1.22	0.84	2	50	1.28	i	m	563	....	....	407	0.72	208	....	....	170	0.82	....	....
	5	....	3+	3+	2	50	3+	m	OK	405	....	....	135	0.33	120	....	....	182	1.52	....	....
Hi-Early	3	....	0.50	0.51	2	50	0.20	n	OK	433	....	....	362	0.84	302	....	....	192	0.64	....	....
LABORATORY No. 19																					
Normal	10	....	0.02	−0.02	....	....	0.03	OK	OK	382	0.001	0.001	470	1.23	158	0.001	0	227	1.44	....	....
	14	....	0.08	0.05	....	....	0.07	OK	OK	393	0.003	0.001	523	1.33	215	0.002	0.001	190	0.90	....	....
	2	....	0.23	0.11	....	....	0.22	OK	OK	487	0.002	0.002	450	0.92	163	0.005	0.003	185	1.13	....	....
	9	....	0.26	0.10	....	....	0.26	OK	OK	513	0.002	0.001	440	0.86	180	0.001	0.002	183	1.01	....	....
	12	....	0.28	0.13	....	....	0.28	OK	OK	632	0.003	0.002	493	0.78	185	0.004	0.002	192	1.04	....	....
Normal	15	....	0.35	....	....	....	0.38	OK	OK	508	0.004	0.004	455	0.90	188	0.003	0.002	183	0.97	....	....
	13	....	0.38	0.18	....	....	0.36	OK	OK	577	0.003	0.002	380	0.66	193	0.004	0.001	190	0.98	....	....
	7	....	0.38	0.16	....	....	0.40	OK	OK	393	0.003	0.004	388	0.98	202	0.001	0.001	170	0.84	....	....
	5	....	0.45	0.21	....	....	0.48	OK	OK	615	0.001	0.002	407	0.66	217	0	0.001	193	0.89	....	....
	8	....	0.76	0.36	....	....	0.78	OK	OK	510	0.006	0.007	387	0.76	170	0.001	0.002	187	1.10	....	....
Hi-Early	1	....	0.23	0.07	....	....	0.23	OK	OK	512	0.005	0	405	0.79	325	0.013	0.006	255	0.78	....	....
	4	....	0.25	0.11	....	....	0.25	OK	OK	607	0.003	0.004	423	0.70	328	0	0	233	0.71	....	....
	3	....	0.31	0.13	....	....	0.31	OK	OK	663	0.001	0.003	500	0.75	330	0.001	0.002	237	0.72	....	....
	6	....	0.36	0.17	....	....	0.38	OK	OK	575	0.002	0.003	467	0.81	358	0	0	237	0.66	....	....
	11	....	0.38	0.15	....	....	0.38	OK	OK	633	0.005	0.003	480	0.76	382	0.004	0.001	238	0.62	....	....

TABLE I.—COOPERATIVE STUDIES OF VARIOUS TYPES OF SOUNDNESS TESTS.—*Continued*

Type of Cement	Reference Mark	A.S.T.M. Normal Consistency, per cent	Autoclave Test of 1 by 1 by 11¼-in. Neat-Cement Bars					Modified Autoclave Soundness Pat	A.S.T.M. Standard Soundness Pat	Neat-Cement Briquets					1:3 Standard Sand Mortar Briquets					LeChatelier Expansion Test	
			Proposed 1938 Method[2]		Modified Molding Procedure[4]					30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	In Boiling Water	5-hr. Autoclave Test
			Expansion, per cent, Bars Cured Moist for		Number of Layers	Total Number of Tamps	Expansion, per cent, Bars Cured Moist for 1 day				Expansion at Waist Line, in.		Tensile Strength, psi.			Expansion at Waist Line, in.		Tensile Strength, psi.			
			1 day	14 days							Width	Thickness				Width	Thickness				
Group No.			1	1A	2	2	2	3	4	5	6	6	6		7	8	8	8		9	9A
										LABORATORY NO. 20											
Normal	3	....	0.53	0.18	....	....	0.54	OK	OK	352	0.053	0.048	433	1.23	183	0.007	0	245	1.34	....	....
	1	....	0.59	0.18	....	....	0.56	OK	OK	410	0.054	0.074	553	1.35	190	0	0.014	250	1.32	....	....
	2	....	0.62	0.19	....	....	0.60	OK	OK	387	0.043	0.103	467	1.21	180	0.003	0.007	228	1.27	....	....
Hi-Early	4	....	0.06	0.03	....	....	0.06	OK	OK	420	0.003	0.006	593	1.41	333	0.003	0.003	410	1.23	....	....
Type B	5	....	0.07	0.03	....	....	0.07	OK	OK	303	0.013	0.003	547	1.80	213	0.010	0.003	263	1.23	....	....
										LABORATORY NO. 21											
Normal	L	....	0.17	0.11	....	....	0.20	OK	OK	427	....	....	540	1.26	220	....	....	193	0.88	....	....
	M	....	0.19	0.06	....	....	0.20	OK	OK	430	....	....	507	1.18	227	....	....	207	0.91	....	....
	N	....	0.20	0.06	....	....	0.20	OK	OK	430	....	....	520	1.21	207	....	....	177	0.85	....	....
	I	....	0.24	0.04	....	....	0.24	OK	OK	367	....	....	500	1.36	230	....	....	210	0.91	....	....
	C	....	0.27	0.09	....	....	0.28	OK	OK	397	....	....	513	1.29	210	....	....	197	0.94	....	....
	P	....	0.31	0.06	....	....	0.32	OK	OK	445	....	....	480	1.08	240	....	....	218	0.91	....	....
	D	....	0.34	0.09	....	....	0.35	OK	OK	367	....	....	437	1.19	203	....	....	190	0.94	....	....
	O	....	0.35	0.09	....	....	0.34	OK	OK	427	....	....	553	1.29	253	....	....	203	0.80	....	....
	B	....	0.37	0.12	....	....	0.47	OK	OK	463	....	....	500	1.08	240	....	....	193	0.80	....	....
	K	....	0.38	0.02	....	....	0.31	OK	OK	383	....	....	553	1.44	217	....	....	187	0.86	....	....
	J	....	0.43	0.19	....	....	0.41	OK	OK	387	....	....	527	1.36	190	....	....	187	0.98	....	....
	G	....	0.47	0.05	....	....	0.47	OK	OK	473	....	....	450	0.95	260	....	....	180	0.69	....	....
	H	....	0.50	0.08	....	....	0.50	OK	OK	417	....	....	540	1.29	240	....	....	170	0.71	....	....
	E	....	0.57	0.16	....	....	0.57	OK	OK	347	....	....	457	1.32	217	....	....	203	0.94	....	....
	A	....	0.61	0.19	....	....	0.60	OK	OK	433	....	....	523	1.21	253	....	....	183	0.72	....	....
	F	....	0.66	0.18	....	....	0.65	OK	OK	443	....	....	457	1.03	277	....	....	200	0.72	....	....
										LABORATORY NO. 22											
Normal	D-1	....	0.00	−0.02	1	100	0.00	OK	OK	468	....	....	501	1.07	170	....	....	236	1.39	....	....
	D	....	0.10	0.01	1	100	0.10	OK	OK	462	....	....	486	1.05	183	....	....	261	1.42	....	....
	C	....	0.37	0.08	1	100	0.38	OK	OK	285	....	....	344	1.21	153	....	....	177	1.16	....	....
	B	....	0.51	0.19	1	100	0.51	OK	OK	555	....	....	533	0.96	153	....	....	201	1.31	....	....
	C-1	....	0.57	0.11	1	100	0.60	OK	OK	314	....	....	327	1.04	135	....	....	194	1.44	....	....
	A-1	....	0.67	0.24	1	100	0.70	OK	OK	374	....	....	387	1.04	165	....	....	185	1.12	....	....
	B-1	....	0.74	0.29	1	100	0.74	[c]	OK	446	....	....	386	0.85	162	....	....	168	1.04	....	....
	B-2	....	1.53	0.14	1	100	1.54	[o]	OK	467	....	....	295	0.63	165	....	....	172	1.04	....	....
	A	....	5.27	0.17	1	100	5.41	[p]	OK	476	....	....	164	0.34	171	....	....	209	1.22	....	....

LABORATORY NO. 23

Normal	....	....	0.10	0.05	1	56	0.09	OK	OK	377	0	0	453	1.20	155	0	0	217	1.40	....	....
Hi-Early	....	....	0.01	−0.03	1	56	0.01	OK	OK	383	0	0	608	1.59	289	0	0	305	1.06	....	....
	....	....	0.27	0.12	1	72	0.29	OK	OK	235	0	0	453	1.92	267	0	0	262	0.98	....	....

LABORATORY NO. 24*

Normal	9	....	0.21	0.13	....	....	0.21	OK	OK	427	....	....	522	1.22	220	....	....	183	0.83	....	....
	6	....	0.23	0.16	....	....	0.23	OK	OK	397	....	....	490	1.23	237	....	....	220	0.92	....	....
	4	....	0.25	0.11	....	....	0.24	OK	OK	397	....	....	445	1.12	235	....	....	207	0.88	....	....
	7	....	0.25	0.09	....	....	0.25	OK	OK	428	....	....	420	0.98	195	....	....	205	1.05	....	....
	11	....	0.32	0.26	....	....	....	OK	OK	482	....	....	447	0.92	200	....	....	162	0.81	....	....
	2	....	0.37	1.29	....	....	0.38	OK	OK	473	....	....	418	0.88	202	....	....	180	0.89	....	....
	8	....	0.47	0.15	....	....	0.47	OK	OK	360	....	....	540	1.50	167	....	....	177	1.06	....	....
	3	....	0.63	0.28	....	....	0.64	OK	OK	462	....	....	370	0.80	178	....	....	173	0.97	....	....
	5	....	0.89	0.38	....	....	0.87	OK	OK	348	....	....	390	1.12	182	....	....	169	0.92	....	....
	10	....	1.66	2.46	....	....	....	OK	OK	517	....	....	388	0.75	197	....	....	170	0.86	....	....
	1	....	1.70	2.20	....	....	1.65	OK	OK	510	....	....	395	0.77	205	....	....	155	0.75	....	....
Dark, Normal	12	....	0.07	0.05	....	....	0.07	OK	OK	465	....	....	515	1.10	187	....	....	183	0.97	....	....
	13	....	0.11	0.08	....	....	0.11	OK	OK	390	....	....	543	1.39	238	....	....	235	0.98	....	....
Old Fash.	15	....	0.20	0.08	....	....	0.20	OK	OK	380	....	....	457	1.20	127	....	....	155	1.22	....	....
	17	....	0.20	0.11	....	....	0.21	OK	OK	415	....	....	505	1.21	143	....	....	145	1.01	....	....
	16	....	0.21	0.11	....	....	0.20	OK	OK	453	....	....	495	1.09	123	....	....	160	1.30	....	....
	14	....	0.22	0.08	....	....	0.22	OK	OK	392	....	....	457	1.16	118	....	....	145	1.23	....	....
Hi-Early	18	....	−0.03	−0.06	....	....	−0.03	OK	OK	508	....	....	447	0.88	323	....	....	253	0.78	....	....
	21	....	0.14	0.10	....	....	0.16	OK	OK	457	....	....	430	0.94	322	....	....	188	0.58	....	....
	19	....	0.15	0.11	....	....	0.16	OK	OK	433	....	....	427	0.98	355	....	....	253	0.71	....	....
	20	....	0.61	0.27	....	....	0.59	OK	OK	442	....	....	443	1.00	330	....	....	192	0.58	....	....

LABORATORY NO. 25

Normal	20	23.0	−0.01	−0.01	1	40	−0.02	OK	OK	325	0.001	−0.001	470	1.44	130	0.002	−0.006	215	1.65	....	....
	15	25.0	0.03	0.00	1	43	0.03	OK	OK	355	0	0.001	370	1.04	150	0.001	0.001	225	1.50	....	....
	16	25.0	0.04	0.01	1	43	0.04	OK	OK	360	0.001	0	380	1.06	180	−0.001	0.001	240	1.33	....	....
	17	25.0	0.04	0.01	1	33	0.04	OK	OK	355	0	0.002	345	0.97	200	0.001	0	205	1.02	....	....
	12	24.0	0.19	0.13	1	40	0.19	OK	OK	330	0.002	0.003	420	1.27	130	−0.002	0.003	160	1.23	....	....
	10	24.5	0.21	0.10	1	42	0.21	OK	OK	345	0.001	0.001	410	1.19	140	0.004	0.001	200	1.43	....	....
	13	24.5	0.22	0.11	1	40	0.21	OK	OK	365	0.003	0.002	445	1.22	100	0.005	−0.008	155	1.55	....	....
	21	24.5	0.24	0.09	1	38	0.24	OK	OK	370	0.003	0.002	440	1.19	155	0	0.001	190	1.23	....	....
Portland-Pozzuolana	4	27.0	−0.02	−0.02	1	38	−0.02	OK	OK	210	0.002	0.001	425	2.02	60	0.001	−0.001	360	6.00	....	....
	3	27.5	0.04	−0.01	1	38	0.04	OK	OK	320	0.001	0	585	1.80	115	0.002	0.001	270	2.35	....	....
	5	27.0	0.04	0.01	1	40	0.04	OK	OK	410	0.001	0.001	505	1.23	195	0.001	0.001	290	1.49	....	....
Special	22	26.5	−0.03	−0.05	1	38	−0.02	OK	OK	280	0	0.001	580	2.07	335	0.001	0.001	335	1.00	....	....
	23	22.0	−0.01	−0.01	1	38	−0.01	OK	OK	380	0	−0.001	645	1.70	130	0.002	0.002	225	1.73	....	....
	25	24.0	0.02	0.02	1	38	0.02	OK	OK	410	0.001	0.002	455	1.11	150	0.003	−0.002	225	1.50	....	....
	24	23.5	0.15	0.11	1	40	0.15	OK	OK	340	0.005	0.003	675	1.98	115	0.001	0.003	195	1.70	....	....
	6	23.0	0.18	0.14	1	38	0.19	OK	OK	455	0.002	0.002	475	1.04	135	0.001	0.002	155	1.15	....	....
	2	25.0	7.21	11.7	1	38	7.21	unsound	OK	515	0.102	0.125	55	0.11	320	0.001	0.001	240	0.75	....	....
	26	28.0	37.5	13.8	1	38	37.5	unsound	unsound	415	0.288	0.315	120	0.29	50	0.208	0.201	15	0.30	....	....

* Laboratory No. 24 used 1 by 1 by 6¼-in. neat bars

TABLE I.—COOPERATIVE STUDIES OF VARIOUS TYPES OF SOUNDNESS TESTS.—*Continued*

Type of Cement	Reference Mark	A.S.T.M. Normal Consistency, per cent	Autoclave Tests of 1 by 1 by 11¼-in. Neat-Cement Bars					Modified Autoclave Soundness Pat	A.S.T.M. Standard Soundness Pat	Neat-Cement Briquets					1:3 Standard Sand Mortar Briquets					LeChatelier Expansion Test	
			Proposed 1938 Method[2]		Modified Molding Procedure[4]					30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	In Boiling Water	5-hr. Autoclave Test
			Expansion, per cent, Bars Cured Moist for		Number of Layers	Total Number of Tamps	Expansion, per cent, Bars Cured Moist for 1 day				Expansion at Waist Line, in.		Tensile Strength, psi.			Expansion at Waist Line, in.		Tensile Strength, psi.			
			1 day	14 days							Width	Thickness				Width	Thickness				
Group No.			1	1A	2	2	2	3	4	5	6	6	6		7	8	8	8		9	9A
LABORATORY NO. 26																					
Normal	4	....	0.03	−0.10	2	....	0.03	OK	OK	445	....	....	410	0.92	250	....	....	205	0.82	....	....
	5	....	0.13	....	2	....	0.13	OK	OK	395	....	....	380	0.96	185	....	....	165	0.89	....	....
	1	....	0.69	0.53	2	....	0.67	OK	OK	400	....	....	350	0.87	215	....	....	180	0.84	....	....
	3	....	1.13	0.22	2	....	1.11	m	OK	520	....	....	210	0.40	220	....	....	190	0.86	....	....
	2	....	2.26	....	2	....	2.23	m	OK	450	....	....	80	0.18	180	....	....	165	0.92	....	....
Hi-Early	7	....	0.02	−0.02	2	....	0.02	OK	OK	425	....	....	615	1.45	315	....	....	225	0.71	....	....
	8	....	0.04	....	2	....	0.04	OK	OK	370	....	....	500	1.35	310	....	....	375	1.21	....	....
	6	....	0.05	0.01	2	....	0.04	OK	OK	340	....	....	655	1.93	340	....	....	360	1.06	....	....
Modified	10	....	0.03	....	2	....	0.03	OK	OK	375	....	....	400	1.07	235	....	....	205	0.87	....	....
	9	....	0.10	0.06	2	....	0.09	OK	OK	365	....	....	565	1.55	250	....	....	230	0.92	....	....
Low Heat	11	....	0.02	0.00	2	....	0.02	OK	OK	310	....	....	485	1.56	90	....	....	180	2.00	....	....
	12	....	0.04	....	2	....	0.04	OK	OK	425	....	....	505	1.19	155	....	....	210	1.36	....	....
Waterproof	14	....	0.13	....	2	....	0.13	OK	OK	350	....	....	460	1.31	195	....	....	220	1.13	....	....
	13	....	0.28	0.16	2	....	0.28	OK	OK	415	....	....	505	1.22	250	....	....	185	0.74	....	....
Blended	15	....	0.00	−0.04	2	....	0.00	OK	OK	400	....	....	515	1.29	265	....	....	255	0.96	....	....
Sulfate-Resisting	16	....	0.02	....	2	....	0.02	OK	OK	385	....	....	410	1.06	185	....	....	235	1.27	....	....
Oil-Well	19	....	−0.10	−0.01	2	....	0.01	OK	OK	175	....	....	395	2.26	65	....	....	170	2.62	....	....
	20	....	−0.02	−0.03	2	....	−0.02	OK	OK	265	....	....	575	2.17	90	....	....	200	2.22	....	....
	17	....	0.01	−0.02	2	....	0.01	OK	OK	315	....	....	485	1.54	260	....	....	210	0.81	....	....
	18	....	0.01	....	2	....	0.01	OK	OK	355	....	....	345	0.97	140	....	....	140	1.00	....	....
LABORATORY NO. 27																					
N.Y. Board Water Supply	I	22.1	−0.02	−0.03	2	50	−0.01	OK	OK	427	0	0	557	1.30	170	0	0	268	1.58	....	....
	H	23.0	−0.01	0.00	2	125	−0.01	OK	OK	397	0	0	440	1.11	170	0	0	230	1.35	....	....
	J	23.7	0.07	0.02	2	50	0.07	OK	OK	413	0	0	443	1.07	145	0	0	265	1.83	....	....
Normal, Dark	E	23.0	0.03	0.04	2	60	0.03	OK	q	525	0	0	487	0.93	238	0	0	255	1.07	....	....

Normal, Light	G	22.8	0.11	0.10	2	60	0.11	OK	OK	423	0	0	430	1.02	197	0	0	207	1.05	....	....
Fine Ground Hi-Early	B	26.5	0.13	0.09	2	50	0.13	OK	OK	492	0	0	497	1.01	382	0	0	313	0.82	....	....
Normal Hi-Early	A	27.0	0.19	0.14	2	75	0.18	OK	OK	407	0	0	352	0.86	333	0	0	310	0.93	....	....
Normal, Light	F	24.0	0.21	0.15	2	90	0.21	OK	OK	557	0.01	0.01	437	0.78	305	0	0	275	0.90	....	....
Poor Normal, Light	C	22.4	0.29	0.23	2	50	0.29	OK	OK	452	0.01	0.01	455	1.01	205	0	0	203	0.99	....	....
Normal, Light	D	23.5	0.33	0.26	2	70	0.32	OK	OK	550	0	0	468	0.85	260	0	0	227	0.87	....	....
Laboratory No. 28																					
Normal	5	....	0.00	−0.02	2	160	0.00	OK	cracks	398	0	0	548	1.38	213	0	0	213	1.00	0 mm.	....
	3	....	0.02	−0.03	2	160	0.02	OK	OK	457	0	0	372	0.81	200	0	0	215	1.07	0 mm.	....
	6	....	0.67	0.25	2	160	0.68	OK	OK	328	1/64	0	430	1.31	188	0	1/64	168	0.89	1 mm.	....
	4	....	0.98	0.40	2	160	0.97	OK	OK	508	0	0	318	0.63	193	0	0	160	0.83	1 mm.	....
	2	....	1.03	0.27	2	160	1.13	c	cracks	407	1/64	1/64	257	0.63	212	0	1/64	182	0.86	1 mm.	....
	1	....	1.27	0.31	3	240	1.22	c	OK	418	1/64	1/64	208	0.50	177	0	1/64	192	1.09	1 mm.	....
Hi-Early	8	....	0.09	0.05	2	160	0.09	OK	cracks	333	0	0	425	1.28	340	0	0	177	0.52	1 mm.	....
	7	....	0.35	0.20	2	160	0.36	OK	cracks	383	0	0	450	1.17	282	0	0	197	0.70	1 mm.	....
White	10	....	0.02	−0.01	2	160	0.02	OK	cracks	420	0	1/64	438	1.04	173	0	0	187	1.08	0 mm.	....
	9	....	0.05	0.01	2	160	0.05	OK	cracks	415	0	0	423	1.02	205	0	0	198	0.97	2 mm.	....
Laboratory No. 29																					
Cement from Clinker Thought or Known to be Underburned	10	....	0.03	−0.02	2	....	0.03	OK	OK	530	....	....	601	1.13	153	....	....	237	1.55	....	....
	7	....	0.04	0.01	2	....	0.05	OK	OK	380	....	....	507	1.34	160	....	....	260	1.62	....	....
	11	....	0.05	....	2	....	0.05	OK	OK	487	....	....	600	1.23	163	....	....	217	1.33	....	....
	8	....	0.07	....	2	....	0.07	OK	OK	355	....	....	537	1.51	151	....	....	293	1.94	....	....
	9	....	0.10	....	2	....	0.10	OK	OK	435	....	....	597	1.16	183	....	....	213	1.37	....	....
	14	....	0.15	....	2	....	0.15	OK	OK	347	....	....	450	1.29	250	....	....	257	1.03	....	....
	6	....	0.16	....	2	....	0.14	OK	OK	440	....	....	443	1.00	103	....	....	183	1.77	....	....
	17	....	0.22	0.00	2	....	0.22	OK	OK	383	....	....	377	0.98	157	....	....	190	1.21	....	....
	12	....	0.26	....	2	....	0.26	OK	OK	453	....	....	513	1.13	250	....	....	250	1.00	....	....
	15	....	0.52	....	2	....	0.62	OK	OK	457	....	....	340	0.74	190	....	....	181	0.96	....	....
	20	....	0.64	....	2	....	0.64	OK	OK	365	....	....	281	0.77	185	....	....	175	0.96	....	....
	19	....	1.22	....	2	....	1.21	r	OK	250	....	....	133	0.53	63	....	....	105	1.66	....	....
	3	....	2.31	0.35	2	....	2.13	OK	OK	457	....	....	203	0.44	303	....	....	150	0.50	....	....
	18	....	2.48	0.45	2	....	2.48	r	OK	295	....	....	133	0.45	100	....	....	147	1.47	....	....
	5	....	7.80	10.2	2	....	7.80	unsound	OK	363	....	....	....	....	237	....	....	....	....	....	....
	4	....	9.09	7.95	2	....	8.54	unsound	OK	470	....	....	....	....	227	....	....	110	0.49	....	....
	13	....	disintegrated	....	2	....	disintegrated	disintegrated	disintegrated	605	....	....	0	0	127	....	....	0	0	....	....
	16	....	disintegrated	....	2	....	disintegrated	disintegrated	disintegrated	510	....	....	0	0	163	....	....	0	0	....	....
Low Heat	1	....	0.03	−0.02	2	....	0.03	OK	OK	383	....	....	573	1.50	93	....	....	187	2.01	....	....
	2	....	0.07	−0.06	2	....	0.06	OK	OK	257	....	....	597	2.32	80	....	....	170	2.13	....	....

TABLE I.-COOPERATIVE STUDIES OF VARIOUS TYPES OF SOUNDNESS TESTS.—*Concluded*

Type of Cement	Reference Mark	A.S.T.M. Normal Consistency, per cent	Autoclave Tests of 1 by 1 by 11¼-in. Neat-Cement Bars					Modified Autoclave Soundness Pat	A.S.T.M. Standard Soundness Pat	Neat-Cement Briquets					1:3 Standard Sand Mortar Briquets					LeChatelier Expansion Test	
			Proposed 1938 Method[2]		Modified Molding Procedure[4]					30-hr. Moist Tensile Strength, psi.	24 hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	30-hr. Moist Tensile Strength, psi.	24-hr. Moist, 5 hr. in Autoclave, 1 hr. in Water			Ratio Strength of Autoclaved to Moist-Cured Briquets	In Boiling Water	5-hr. Autoclave Test
			Expansion, per cent, Bars Cured Moist for		Number of Layers	Total Number of Tamps	Expansion, per cent, Bars Cured Moist for 1 day				Expansion at Waist Line, in.		Tensile Strength, psi.			Expansion at Waist Line, in.		Tensile Strength, psi.			
			1 day	14 days							Width	Thickness				Width	Thickness				
Group No.			1	1A	2	2	2	3	4	5	6	6	6		7	8	8	8		9	9A
LABORATORY NO. 30																					
Normal	19	....	0.13	....	1	100	0.13	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	20	....	0.15	....	1	100	0.15	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	21	....	0.17	....	1	100	0.18	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	18	....	0.19	....	1	100	0.18	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	22	....	0.19	....	1	100	0.19	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	15	....	0.19	....	1	100	0.20	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	10	....	0.21	....	1	100	0.21	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	12	....	0.21	....	1	100	0.22	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	16	....	0.22	....	1	100	0.22	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	11	....	0.23	....	1	100	0.22	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	14	....	0.23	....	1	100	0.23	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	23	....	0.23	....	1	100	0.23	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	17	....	0.24	....	1	100	0.23	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	13	....	0.26	....	1	100	0.25	....	....	....	....	....	....	....	....	....	....	....	....	....	....
	24	....	0.26	....	1	100	0.26	....	...	....	....	....	....	....	....	....	....	....	....	....	....

[a] Checked.
[b] Slightly warped and checked.
[c] Slightly warped.
[d] Curved, cracked.
[e] Soft, curved, and cracked.
[f] Heavy efflorescence.
[g] Slight efflorescence.
[h] White dust.
[i] Cracks.
[j] Soft, checked.
[k] Distorted.
[l] Fine cracks.
[m] Warped, cracked.
[n] Dry O.K.—wet cracks.
[o] Warped.
[p] Badly warped.
[q] Fine laitance, crazing.
[r] Weak, crumbly—no cracks.

marked 1 tested at the same laboratory. Since little or no data were submitted regarding the cements which showed the unusual results obtained in the group 1A tests, no attempt has been made to explain them at this time.

TABLE II.—CEMENTS HAVING APPRECIABLY HIGHER AUTOCLAVE EXPANSIONS AT 14 DAYS THAN AT 1 DAY.

Laboratory	Reference Mark	Autoclave Expansion, per cent		Modified Autoclave Pat	A.S.T.M. Standard Pat	Remarks
		1 day	14 days			
No. 12	1	0.18	0.48	OK	OK	High early strength
No. 5	7348	0.19	2.1+	OK	OK	Normal
No. 24	2	0.37(0.32)[a]	1.29(0.26)[a]	OK(OK)[a]	OK(OK)[a]	Normal
No. 5	7349	0.40	9.7	OK	OK	High early strength
No. 15	5	0.56	0.65	OK	OK	High early strength
No. 6	8	0.85	3.25	OK	OK	Normal
No. 6	6	1.30	1.60	OK	OK	Normal
No. 24	1	1.70(1.66)[a]	2.20(2.46)[a]	OK(OK)[a]	OK(OK)[a]	Normal
No. 1	C	2.10	8.94	OK	OK	Special
No. 12	5	2.17	2.50	Warped and cracked	OK	Normal
No. 1	D	2.51	3.57	OK	OK	Special
No. 6	2	2.70	Too big	Sound but cracked	OK	Normal
No. 6	9	3.30	Too big	Checked	OK	Normal
No. 25	2	7.21	11.7	Unsound	OK	High early strength
No. 29	5	7.80	10.2	Unsound	OK	Underburned

[a] Check tests on same cement.

Effect of Molding Procedure on Autoclave Expansion.—Of the 263 cements tested in group 2 for comparison with group 1, all but three cements showed substantially the same autoclave expansions when the test bars were molded in one or two layers with as few as 50 tamps, as when they were molded in three layers in accordance with the 1938 proposed method[2] using 150 tamps. These three cements were as follows:

Laboratory	Reference Mark	Autoclave Expansion at 1 day, per cent	
		1938 Proposed Method[2] (Group 1)	Modified Procedure[4] (Group 2)
No. 6	8	0.85	1.12
No. 12	2	0.71	1.64
No. 18	6	0.95	1.29

Since only in the three cases indicated above did the modified procedure give results differing materially from those obtained with the 1938 method in tests on 263 different cements, it may be concluded that the method of molding the test bar is not an important variable so long as the mold is completely filled and a homogeneous specimen is obtained. In filling the mold, it is important that care be exercised to insure that the paste completely surrounds the reference points at the ends of the bar.

Comparison of Autoclave Test with Two Types of Pat Tests.—The data submitted permit some comparisons to be made of the expansions of neat bars in the autoclave test with the behavior of companion modified neat pats in the autoclave test, and with standard neat pats in the present A.S.T.M. steam test.

Both sound, and checked, cracked or warped autoclaved pats were reported for cements showing autoclave expansions at 1 day of from 0.18 to 4.2 per cent. It was not clear from the comments submitted with the test data, whether the checks and cracks noted were significant and of such a nature as to indicate actual unsoundness of the cement. In a few cases the autoclaved pats were reported as having superficial cracks on the top surface, perhaps due to troweling (laboratory No. 18). No sound autoclaved pats were reported for cements having an autoclave expansion above 4.2 per cent at 1 day.

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[2] See Proposed Method of Test for Autoclave Expansion of Portland Cement, *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 297 (1938).

[3] 1937 Supplement to Book of A.S.T.M. Standards, p. 54.

[4] Tentative Method of Test for Autoclave Expansion of Portland Cement (C 151 - 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 176.

Standard pats were reported as showing cracks in the A.S.T.M. soundness test for 9 cements having autoclave expansions less than 0.5 per cent at 1 day. Some of these pats had only superficial cracks in the troweled surface and hence may be discounted. A warped, standard pat was reported for a cement having an autoclave expansion of 1.22 per cent at 1 day, and disintegrated or otherwise unsound standard pats were reported for 3 cements; one of these had an estimated autoclave expansion of 37.5 per cent and the other two disintegrated in the autoclave. Five cements with autoclave expansions above 5 per cent were reported as showing sound standard pats.

TABLE III.—COMPARISON OF TWO TYPES OF PAT TESTS WITH AUTOCLAVE EXPANSIONS.

Autoclave Expansion at 1 day, per cent	Autoclaved Modified Pat						A.S.T.M. Standard Pat					
	Number of Cements Reported	Number of Cements Reported as					Number of Cements Reported	Number of Cements Reported as				
		OK	Cracked	Checked	Warped	Unsound		OK	Cracked	Checked	Warped	Unsound
0.50 or less	216	210	4[a]	2	0	0	216	207	9[d]	0	0	0
0.51 to 1.00	34	30	1[b]	0	3[c]	0	34	34	0	0	0	0
1.01 to 5.00	30	13	2	3	10	2	34	32	1[e]	0	1	0
Over 5.00	8	0	0	0	2	6	8	5	0	0	0	3
Total	288	253	7	5	15	8	292	278	10	0	1	3

[a] Pats for 2 of the 4 cements were reported to be OK when dry but to show a few cracks in the troweled surface when wet; the other 2 cements were reported to have a few cracks in the top surface when examined either wet or dry.

[b] Pat reported OK when dry and to have cracks in troweled surface when wet.

[c] Pats for 2 of the 3 cements were reported as only slightly warped.

[d] Pats for 5 of the 9 cements were reported as showing cracks only after wetting; for two of the cements the cracks were in the troweled surface.

[e] Cracks noted only when pat was wetted.

The results for both types of pat test with respect to the autoclave expansion of parallel neat bars are summarized in Table III.

Perhaps the most significant feature of the data in Table III is that for autoclave expansions above 1 per cent, an appreciable number (34 per cent) of the autoclaved modified pats, and nearly all (88 per cent) of the standard pats, were reported OK. It is unfortunate that the cooperating laboratories were unable to obtain more cements having high autoclave expansions so that a better evaluation of the modified autoclave pat test could be made.

Strength and Volume Change of Briquets Subjected to Autoclave Test.—The ratios of the strengths of autoclaved briquets to the strengths of companion moist-cured briquets at 30 hr. varied over a wide range. This was particularly true for the cements reported as showing an autoclave expansion of 0.5 per cent or less. The following tabulation gives the distribution of the cements with respect to both the autoclave expansions at 1 day, and the ratio of the strengths of the autoclaved briquets to the strengths of the corresponding moist-cured briquets at 30 hr.:

Autoclave Expansion at 1 day, per cent	Number of Cements				
	Total Reported	Ratio of Strength of Autoclaved Briquets, to Strength of Companion Moist-Cured Briquets at 30 hr.			
		0.75 or Less	0.76 to 1.00	1.01 to 1.25	Over 1.25
NEAT BRIQUETS					
0.5 or less	223	18	63	88	54
0.51 to 1.00	33	6	16	8	3
1.01 to 5.00	29	18	6	2	3
Over 5.00	7	7	0	0	0
1:3 STANDARD SAND BRIQUETS					
0.5 or less	202	14	65	55	68
0.51 to 1.00	33	5	14	5	9
1.01 to 5.00	25	5	9	3	8
Over 5.00	6	5	0	1	0

These data indicate that the strength ratio, for both neat and standard mortar briquets, tends to decrease as the autoclave expansion increases, although in numerous cases the results are inconsistent with this general trend.

Figure 1, which was submitted by laboratory No. 29, shows relationships between the autoclave expansion of neat bars and the strength ratios for neat and standard mortar briquets. In this figure most of the plotted points lie on or near the curve. However, two of the cements, Nos. 18 and 19, which were in a group prepared from clinkers reported as purposely underburned, showed high strength ratios in the standard mortar briquets, even though the autoclave expansions were relatively high. The high strength ratios in these two cases may be the result of chemical reaction between certain of the constituents of the cements and the Ottawa silica sand.

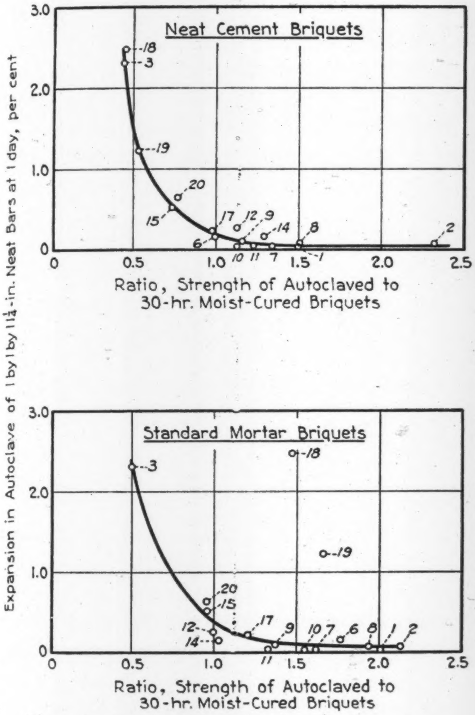

Fig. 1.—Relationship between Expansion in Autoclave of Neat-Cement Bars and of Strength Ratios of Neat-Cement Briquets and of Standard Mortar Briquets.

Submitted by laboratory No. 29. Cement numbers are indicated opposite plotted points (see Table I).

When the data from all of the laboratories are plotted in the same manner as in Fig. 1, there is considerable scattering of the individual points, especially in the case of cements which showed less than about 1 per cent autoclave expansion. Furthermore, high strength ratios were obtained for the standard mortar briquets of cements which had autoclave expansions considerably above 1 per cent. Considering

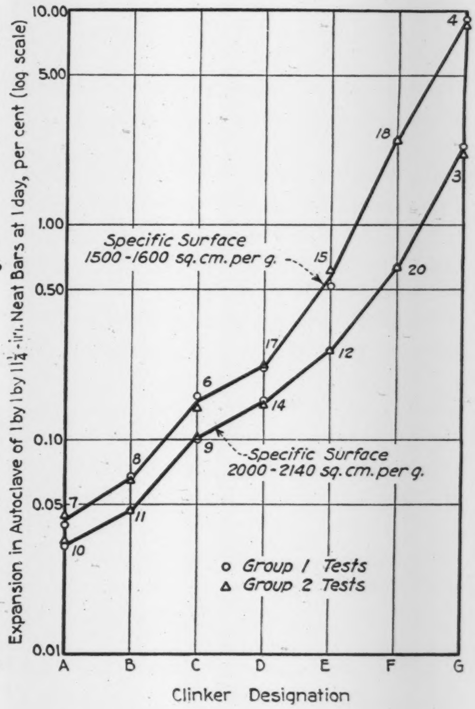

Fig. 2.—Effect of Fineness of Cement on Expansion in Autoclave of Neat-Cement Bars.

Submitted by laboratory No. 29. Cement numbers are indicated opposite plotted points (see Table I).

all the results submitted, it appears that the strength ratios of neat and mortar briquets bear only a general relation to the expansions which occur during the autoclave test and hence are of doubtful value as criteria of soundness.

A study was made of the dimensions of the briquets at the waist line before and after the autoclave test. It was found that the expansions of the neat briquets paralleled somewhat the expansions of the neat bars. On the other hand, the expansions of the standard mortar briquets did not always parallel the expansions of the neat bars. The reliability of such measurements on briquets is questionable because of their relatively small size and also because in some cases the measuring equipment used was not suitable for the purpose.

Volume Change of LeChatelier Test Specimens.—Study of the limited data reported shows no consistent relationship between the LeChatelier expansion during 3 to 5 hr. in boiling water and the autoclave expansion of corresponding neat-cement bars. However, when the LeChatelier specimens were tested in the autoclave, the expansions were about in direct proportion to the expansions of the neat-cement bars tested in the autoclave.

Effect of Fineness of Cement on Autoclave Expansion.—In Fig. 2 are plotted the expansions of neat bars in the autoclave for 14 of the cements reported by laboratory No. 29. These cements were ground in the laboratory from 7 clinkers to two finenesses as indicated in the diagram. It will be noted that in each case the expansion of neat bars in the autoclave was appreciably less for the more finely ground cement; the higher the autoclave expansion of the coarser ground cement, the greater was the reduction in expansion due to finer grinding.

SUMMARY OF RESULTS

1. For 206 (94 per cent) of 221 cements tested, the autoclave expansions of neat bars at 14 days were either approximately the same or were less than the expansions of companion neat bars at 1 day. Of the 15 cements which showed appreciably higher expansions at 14 days than at 1 day, four had less than 1 per cent expansion when autoclaved at 1 day but significantly higher expansions when autoclaved at 14 days. The reasons for this behavior are not clear from the information submitted.

2. The method used in molding the 1 by 1 by $11\frac{1}{4}$-in. neat bars did not affect the 1-day autoclave expansion appreciably. It may be concluded, therefore, that the method of molding is not an important factor, provided the mold is completely filled and homogeneous specimens are obtained.

3. From the data submitted it appears that sound autoclaved pats may be obtained with cements which show autoclave expansions up to 4 or 5 per cent.

Pats in the A.S.T.M. steam test were reported as sound for nearly all of the cements except those having excessively high autoclave expansions at 1 day. The results confirm those of many other tests in showing the inadequacy of the standard pat test as a test for soundness of cement.

4. Ratios of the strengths of autoclaved neat and standard mortar briquets to the 30-hr. strengths of companion moist-cured briquets, showed a general trend toward lower values as the autoclave expansions of the cements increased. However, there were many exceptions to this general trend, indicating that these strength ratios are not very reliable criteria of cement soundness.

5. The limited results reported showed little agreement between the LeChatelier

expansion tests made in boiling water and the autoclave expansions of the cements. When the LeChatelier specimens were subjected to the autoclave test, however, the resulting expansions were roughly proportional to the autoclave expansions of the corresponding neat-cement bars.

6. Tests of a group of 7 clinkers, each ground to two different finenesses showed an appreciable reduction in autoclave expansion for the more finely ground cement; the greatest reductions were obtained with the clinkers showing the highest autoclave expansions.

Respectfully submitted on behalf of the Working Committee,

H. F. GONNERMAN,
Chairman.

REPORT OF WORKING COMMITTEE ON METHODS OF CHEMICAL ANALYSIS

The Working Committee on Methods of Chemical Analysis recommends for adoption as standard the analytical procedures for determining sodium oxide, potassium oxide, water-soluble alkali, phosphorus pentoxide, and manganic oxide in cement as described in Sections 1 to 9, inclusive, of the Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 39 T).[1] Many analysts have obtained very satisfactory results with the use of these procedures and the Working Committee has received no criticism of them. Only two minor changes in the procedure are recommended as follows:

Section 2 (b).—Add a parenthetical reference to "Note" between the fifth and sixth sentences and include the following note:

NOTE.—If it is desired to speed up the work without allowing the residue to settle so as to give a clear solution to decant easily, filter at once the solution leaving as much of the residue in the dish as possible, discharge the filtrate held in the stem of the funnel by tapping the funnel or lifting the filter paper a little out of the funnel, and wash the residue on the filter paper back into the dish.

Section 5.—Change the first eight sentences from their present form to read as follows:

5. Weigh 1 to 3 g. of the sample (Note 1) and 10 g. of NH_4NO_3 into a 150-ml. beaker. Mix the contents, treat with 10 ml. of HNO_3 (sp. gr. 1.42), and stir quickly, using the flattened end of a glass rod to crush lumps of cement, until the cement is completely decomposed and the thick gel of SiO_2 is broken up. Cover the beaker with a watch glass, place it on a water bath or a hot plate at the approximately same temperature for 15 to 20 min., and stir the contents occasionally during the heating. Add 20 ml. of hot water to the beaker and stir the contents. If the cement contains an appreciable amount of manganese as shown by the presence of a red or brown residue, add a few milliliters of $NaNO_2$ solution (5 per cent) to dissolve this residue. Boil the contents of the beaker until all nitrous fumes are completely expelled. This should not take more than 5 min., and water should be added to replace any lost by evaporation. Filter into a 500-ml. Erlenmeyer flask under suction and with a platinum cone to support the filter and wash the residue of SiO_2 until the volume of filtrate and washings is about 150 ml.

It is also recommended that with the adoption of the above mentioned procedures as standard there be included the following recommended order for reporting the minor components in a chemical analysis of portland cement, this to be incorporated in Section 21 (*a*) of the Standard Methods C 114 - 39[2] between the recommended order for reporting major components and the separate determinations:

Minor Components:

Na_2O	Sodium oxide
K_2O	Potassium oxide
P_2O_5	Phosphorus pentoxide
Mn_2O_3	Manganic oxide

The procedures for determining free calcium oxide in portland cement and clinker described in Sections 10 to 13 of Tentative Methods C 114 - 39 T[1] are being continued as tentative pending their further consideration by the committee.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 883.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 8.

The Working Committee also recommends new tentative methods for determining silicon dioxide, calcium oxide, and magnesium oxide[3, 4] for inclusion in Tentative Methods C 114 - 39 T.

Appended to this report the committee is submitting for publication as information proposed methods for determining acid-soluble sodium oxide and potassium oxide and water-soluble alkali in portland cement.

Investigation of Quick Methods for Phosphorus Pentoxide.—Reference has been made in previous reports in regard to the continuation of the studies of the quick volumetric determination of phosphorus pentoxide. Last year both Prof. W. A. Van Winkle of Kansas State College of Agriculture and Applied Science, and Mr. O. A. Ellingson of Universal Atlas Cement Co. developed new modifications of their methods. Ten laboratories have just completed a cooperative series of tests of the methods and the results will be reported to Committee C-1 at the June meeting.

Vinsol Resin and Tallow.—Under the leadership of Mr. T. A. Hicks, General Chemist of the Universal Atlas Cement Co., a member of the Working Committee, a few laboratories carried on some preliminary work with a method for determining vinsol resin and tallow in cement. The results were not quite satisfactory. Another method was then tried but it did not prove to be any better than the first method. The work was continued on a third method by the same group of laboratories and the results were good. A report on this work will be presented in detail to Committee C-1 at this annual meeting.

Respectfully submitted on behalf of the Working Committee,

W. C. HANNA,
Chairman.

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[3] These methods were accepted as tentative by the Society and have been incorporated in Tentative Methods C 114 which appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 171.

[4] For a report on the investigation of these new methods, see W. C. Hanna, L. N. Bryant, and T. A. Hicks, "Cooperative Study of Quick Methods of Determining Silicon Dioxide, Calcium Oxide, and Magnesium Oxide in Portland Cement," published in ASTM BULLETIN, No. 103, March, 1940, p. 29.

APPENDIX

PROPOSED METHOD OF CHEMICAL ANALYSIS OF PORTLAND CEMENT[1, 2]

ACID-SOLUBLE SODIUM OXIDE AND POTASSIUM OXIDE, WATER-SOLUBLE ALKALI

These are proposed methods and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

ACID-SOLUBLE SODIUM OXIDE AND POTASSIUM OXIDE[3]

Procedure

1. (*a*) Place 5 g. of the sample in a casserole and add enough water to make a thin grout. Add enough HCl to decompose the cement completely and evaporate the solution to dryness on a water bath. Add about 150 ml. of water to the residue, digest it on the water bath or a hot plate for 30 min., and then heat it to boiling. Add a few drops of phenolphthalein indicator to the solution and treat it with CaO or $Ca(OH)_2$ until the solution is strongly alkaline. Boil the mixture for a few minutes, allow the residue to settle, and decant the clear solution through a filter paper into a large beaker. Add 30 ml. of water to the casserole, bring the mixture to a boil, allow the residue to settle, and decant the clear filtrate through the same filter paper. Perform three more times this addition of water and decantation, keeping the mixture strongly alkaline all the time by adding more CaO or $Ca(OH)_2$, if necessary. Finally, wash the residue into the filter paper, rinse the casserole, and wash the residue once with hot water.

(*b*) Follow the procedure as described in Section 21 (*c*) to (*f*) of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114) of the American Society for Testing Materials.[4]

WATER-SOLUBLE ALKALI[5]

Reagents

2. *Standard Hydrochloric Acid* (*0.5 N*).—Prepare a 0.5 *N* HCl solution and standardize it against an acidimetric standard furnished by the National Bureau of Standards (No. 39, benzoic acid; or No. 84, acid potassium phthal-

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[1] These proposed methods are under the jurisdiction of the A.S.T.M. Committee C-1 on Cement.

[2] These proposed methods, while in use, have not been formally adopted. When formally approved, they will be incorporated in the Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 171.

[3] This method is applicable also to the determination of total sodium oxide and potassium oxide if the insoluble residue in cement is small and does not contain an appreciable amount of Na_2O and K_2O.

The decomposition of cement by treating the sample with HCl and later with CaO, instead of igniting the cement with NH_4Cl and $CaCO_3$ as in the method for determining total Na_2O and K_2O described in Sections 1 and 2 of the Tentative Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114),[4] was suggested by a method received from F. H. Jackson Senior Engineer of Tests, Public Roads Administration.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 8.

[5] This procedure is based on the test for free alkali which appeared in the specifications prepared by the late Thaddeus Merriman, Consulting Engineer for the Board of Water Supply of the City of New York. The specifications were published in *Rock Products*, Vol. 41, No. 1, p. 86, January, 1938. An improved procedure was given in a private communication by N. T. Stadtfeld, Assistant Engineer of the Board of Water Supply. The improvement was the result of suggestions made by Nathan Goodman, Senior Chemist of the Board of Water Supply, and O. A. Ellingson, Chief Chemist of Universal Atlas Cement Co., Duluth, Minn.

ate) through a solution of NaOH according to the directions furnished with the standard. If the solution is exactly 0.5 *N* HCl, each 1 ml. of it is equivalent to 0.01550 g. of Na_2O, and, when used according to the procedure described below, is equivalent to 0.00969 per cent Na_2O.

Procedure

3. (*a*) Place 800 g. of the sample (Note 1) in an enameled saucepan and add 500 ml. of distilled water. Stir the slurry every 15 min. for 2 hr. and then filter it through a large folded filter paper. Measure 100 ml. (Note 2) of the filtrate into a beaker. Add to the solution 30 ml. of $Ba(OH)_2$ (saturated solution at room temperature) or more if necessary to precipitate all the sulfate ions that may be present. Heat the solution for 15 min. or longer (Note 3), filter, and wash the residue five times with hot water. Add one or two drops of phenolphthalein indicator (1 per cent in ethanol) to the filtrate and pass CO_2 (Note 4) through it until the pink color disappears. Without filtering, heat the solution until the pink color reappears (Note 5). Filter the solution and wash the residue five times with hot water. Evaporate the filtrate and washings to about 50 ml., filter the solution, and wash the filter paper and residue, if any, with water. Titrate the filtrate and washings at room temperature with 0.5 *N* HCl in the presence of a few drops of methyl orange indicator, which is most conveniently added after the disappearance of the pink color of phenolphthalein. Calculate as Na_2O (Note 6) the percentage of water-soluble alkali in the sample by multiplying the number of milliliters of the standard acid required for the neutralization by 0.00969.

NOTE 1.—The procedure is based on the assumptions that alkali in cement dissolves in water only as NaOH, KOH, Na_2SO_4, and K_2SO_4 and that no other compounds are present which cannot be removed under the prescribed conditions. $Ba(OH)_2$ is used to convert sulfates to hydroxides, and excess $Ba(OH)_2$ is removed along with $Ca(OH)_2$ by the treatment with CO_2. Finally, the alkali, as carbonates and bicarbonates, is titrated with standard acid. The procedure is applicable to most samples of portland cement, but not to cement that contains soluble admixtures such as chlorides and nitrates.

NOTE 2.—If the slurry is so slimy that 100 ml. of filtrate cannot be obtained, all that can be obtained may be used. The volume of the filtrate may be increased by gently tapping the funnel, containing the cement on the folded filter paper, with the palm of the hand, and also by letting the cement settle in the saucepan for about 5 min. before proceeding with the filtration. When the content of water-soluble alkali is large and greater speed is desired for routine testing, a volume of the filtrate smaller than 100 ml. may be taken for the determination. When the volume is less than 100 ml., use a correspondingly smaller amount of $Ba(OH)_2$ solution (sufficient to precipitate all the sulfate ions that may be present), and multiply the volume of standard acid required for neutralization by 100 and divide it by the volume of the original filtrate prior to the calculation of the percentage of Na_2O.

NOTE 3.—The solution may be allowed to stand 1 hr. or longer, and filtered without heating, but heating is desirable as it makes the filtration and washing more rapid. It is advisable to boil the solution if it can be done without bumping. It may be filtered immediately after boiling for 1 or 2 min. If desired, the solution may be heated to boiling before the addition of the $Ba(OH)_2$ solution which may be added either cold or hot.

NOTE 4.—Carbon dioxide may be obtained from a tank of liquid CO_2 or a flask containing pure lump $CaCO_3$ on which HCl (1:1) is dropped. The gas should be passed through a gas washing bottle which contains a saturated solution of $Ca(HCO_3)_2$. The solution may be prepared by putting a saturated solution of $Ca(OH)_2$ in the bottle. Most of the calcium will be precipitated as $CaCO_3$ but a little of it will be redissolved. The excess $CaCO_3$ should be allowed to stay in the bottle. The solution may be replaced by a saturated solution of $NaHCO_3$ if the bottle is so designed that there is no danger of any of the solution being carried over.

NOTE 5.—The reappearance of the pink color may be hastened by boiling the solution, if bumping can be avoided. If the filtration is made before the pink color reappears or just when it begins to show, an appreciable amount of $CaCO_3$ may be precipitated when the clear filtrate is heated. If the solution is boiled a few minutes after the reappearance of the pink color prior to filtration, the calcium is completely precipitated, except probably a slight amount that may show up when the filtrate is evaporated down to about 50 ml.

NOTE 6.—Both Na_2O and K_2O are generally present in any sample of cement, but it is usually considered satisfactory to assume all alkali to be Na_2O.

(*b*) If it is required that water-soluble alkali be reported as water-soluble Na_2O and water-soluble K_2O, use the neutralized solution (Note 7) obtained from the titration and follow the procedure as described in Section 21 of the Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114) of the American Society for Testing Materials[4], starting with Paragraph (*e*) and following all details except that the washing of the precipitate of K_2PtCl_6 by decantation with the Gladding wash is omitted and the precipitate is transferred to the crucible with the ethanol instead of the Gladding wash (Note 8).

NOTE 7.—The neutralized solution theoretically represents the water-soluble alkali in 160 g. of cement when 100 ml. of the original filtrate is used. The error due to the combination of water with cement in the slurry or the possible adsorption of alkali by the cement particles is not appreciable in most cases. If desired, a measured portion of the neutralized solution may be taken in order to avoid the use of a large amount of chloroplatinic acid.

NOTE 8.—The Gladding wash is used to remove any Na_2SO_4 that may contaminate the precipitate of K_2PtCl_6, but in this method sodium and potassium are present as chlorides instead of sulfates.

(*c*) Calculate the percentages of Na_2O and K_2O in the sample as follows:

$$\text{Wt. of } K_2SO_4 = \text{wt. of } K_2PtCl_6 \times 0.3584$$

$$\text{Wt. of } Na_2SO_4 = \text{wt. of } (Na_2SO_4 + K_2SO_4) - \text{wt. of } K_2SO_4$$

$$Na_2O\text{, per cent} = \frac{\text{wt. of } Na_2SO_4 \times 0.4364}{\text{wt. of sample}} \times 100$$

$$K_2O\text{, per cent} = \frac{\text{wt. of } K_2PtCl_6 \times 0.1937}{\text{wt. of sample}} \times 100$$

REPORT OF COMMITTEE C-4

ON

CLAY PIPE

Committee C-4 on Clay Pipe held one meeting during the year: in Chicago, Ill., on April 9, 1940.

Subsequent to the 1938 annual meeting the committee presented to the Society, through Committee E-10 on Standards, a proposed tentative revision of the Standard Specifications for Clay Sewer Pipe (C 13 – 35), which provides for the adoption of a higher minimum crushing strength for clay pipe. This revision was accepted[1] for publication as tentative on August 25, 1938, and appears in the 1939 Book of A.S.T.M. Standards.[2]

Since this revision has been published as tentative for over a year, the committee now recommends that it be approved for reference to letter ballot of the Society for adoption and incorporation in the present standard.[3]

The committee recommends that the Tentative Definitions of Terms Relating to Clay Sewer Pipe (C 8 – 35 T)[4] be withdrawn by the Society, since the committee is of the opinion that the present tentative definitions serve no particularly useful purpose so far as the industry is concerned.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 23 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Spec. for Clay Sewer Pipe (C 13 - 35)[a]	18	3	1
II. WITHDRAWAL OF TENTATIVE STANDARD			
Definitions of Terms Relating to Clay Sewer Pipe (C 8 - 35 T)	21	0	1

[a] The classified vote on the adoption of the tentative revision of the Standard Specifications for Clay Sewer Pipe (C 13 - 35) was as follows: Affirmative, 8 producers, 5 consumers, 5 general interests; negative, 2 producers, 1 consumer.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 23 members; 20 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

ANSON MARSTON,
Chairman.

R. G. SCOTT,
Secretary.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee C-4 reported results of the letter ballot vote as follows: Of a total membership of 20, 18 members returned their ballots, of whom 15 voted affirmatively, 2 negatively, and 1 member marked his ballot "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 1181.

[3] *Ibid.*, p. 236.

[4] *Ibid.*, p. 983.

EDITORIAL NOTE

Subsequent to the annual meeting Committee C-4 presented to the Society through Committee E-10 on Standards a tentative revision of the Standard Specifications for Clay Sewer Pipe (C 13 – 40). This revision was accepted by Committee E-10 for publication as tentative on August 28, 1940, and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 344.

REPORT OF COMMITTEE C-5
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTION

Meetings of Committee C-5 on Fire Tests of Materials and Construction were held on March 28, 1940, in New York City, and on June 26, 1940, in Atlantic City.

I. New Tentative Standard

After several years consideration, interrupted by the death of Prof. A. H. Beyer of Columbia University, former chairman of Subcommittee III, Methods of Fire Tests of Door Assemblies have been prepared and are presented for publication as tentative.[1]

The need for such methods has been felt for some years, having been first expressed at the June, 1930, meeting when it was voted to appoint a Subcommittee on Methods of Test for Wall Opening Protectives. Recent legislation, more especially that of New York State, fixing definite fire-resistance requirements for doors in multifamily residence buildings, further emphasized this need.

The scope of the work of Subcommittee III is broad, covering not only doors but windows and other forms of protection against the spread of fire and other hazards. Because of widely varying requirements that opening protectives are required to meet, the physical nature of the different materials (steel, wood, glass, etc.) entering into the construction of protectives, and the more or less complicated make-up of the complete assembly, it soon became apparent that the efforts of the subcommittee should be directed at first to a limited phase of the subject under consideration. It was therefore decided that the subcommittee devote its attention for the time being to the preparation of methods of fire tests of doors. It then developed that, while reasonable unanimity regarding fire endurance and heat transmission qualities was attainable, the questions involved in the matters of smoke generation and of the creation and spread of dangerous fumes presented problems for the solution of which more time and study are necessary. It is the opinion of Committee C-5, as represented at the March meeting, that on the recommendation of Subcommittee III on Fire Tests of Wall Opening Protectives (A. L. Brown, chairman) the proposed test method[1] should be submitted at this time for publication as tentative, subject to later amendments with respect to fumes and smoke.

II. Revision of Tentative Standard

On the recommendation of Subcommittee VI on Fire Tests of Scaffolding (W. B. White, chairman), the Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 – 37 T)[2] are being revised as follows and continued as tentative:

Section 6.—Change from its present form: namely,

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[1] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 194.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 951.

> 6. Specimens for the timber test shall be $\frac{3}{4}$ in. in thickness, $1\frac{1}{2}$ in. in width, and 10 in. in length. Each specimen shall be taken from the edge of, and not nearer than 2 ft. from, the end of each sample selected for test, and shall be carefully cut from the narrow face of the sample.

to read as follows:

> 6. (*a*) Specimens for the test, commonly known as the timber test, shall be $\frac{3}{4}$ in. in thickness, 10 in. in length, and of a width equal to the least dimension of the sample except as hereinafter specified. Specimens shall be cut to exact dimensions 2 ft. or more from either end of the sample selected as shown in Fig. 1(*a*).

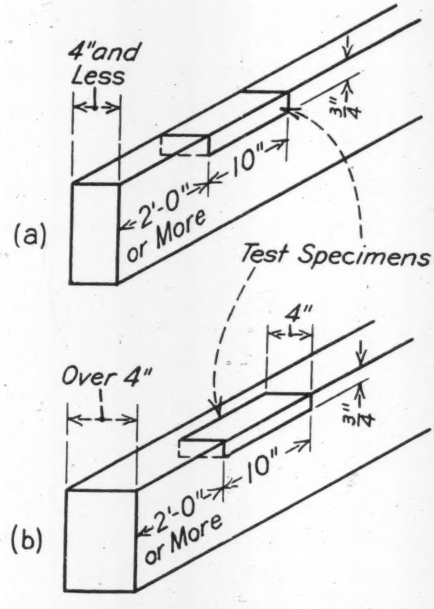
4"and Less
$\frac{3}{4}$"
10"
2'-0" or More
(a)
Test Specimens
4"
Over 4"
$\frac{3}{4}$"
10"
2'-0" or More
(b)

Fig. 1.—Method of Cutting Test Specimens.

> (*b*) When the least dimension of the sample is more than 4 in., test specimens $\frac{3}{4}$ in. in thickness, 4 in. in width, and 10 in. in length shall be cut from each sample to provide two test specimens with one uncut $\frac{3}{4}$-in. edge as shown in Fig. 1(*b*).
>
> (*c*) When samples are selected from tongued and grooved material the test specimens shall be cut from the grooved edge.

Section 9.—Change the first sentence to read as follows by the addition of the italicized words:

> Two weighed test specimens shall be placed side by side across the top of a 6-in. gas crucible furnace with the uncut faces down and their narrow uncut edges together *as shown in Fig. 3* (the accompanying Fig. 2).

Experience in the application of the present tentative specifications has made this proposed revision desirable. Scaffolding and shoring materials are of varying dimensions depending on the purposes for which they are to be used, such as planks, joists, posts, etc. The specifications do not contemplate a complete impregnation of these materials, but merely enough to guard against the hazards of hot rivets, lighted cigarettes, etc. When the thinnest dimension of such material is more than $1\frac{1}{2}$ in., test specimens cut to meet the present specifications, placed over the 6-in. crucible furnace, must of necessity

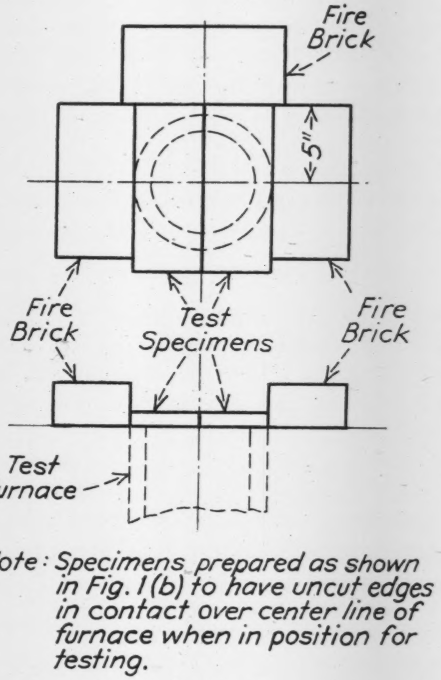
Fire Brick
5"
Fire Brick
Test Specimens
Fire Brick
Test Furnace
Note: Specimens prepared as shown in Fig. 1 (b) to have uncut edges in contact over center line of furnace when in position for testing.

Fig. 2.—Arrangement of Test Specimens and Fire Brick Over Test Furnace.

expose at least two sides that have not been impregnated to the same extent as the outer exposed faces of the material. When placed over the crucible, at least one untreated side is directly exposed to the heat, making the results not comparable to those of specimens taken from the edges of thinner material. By permitting the specimens to be 4 in. wide the untreated edges would be just beyond the opening of the furnace, and only treated surfaces would be exposed to the direct heat of the furnace. Obviously greater uniformity in testing and in results would be obtained.

Partially impregnated fire-retardant wood is, relatively speaking, a new product. For interior trim of buildings complete impregnation or saturation treatments are essential since such wood must be worked up into various shapes and sizes. For structural timber used for scaffolding no such requirement exists and a more limited impregnation gives ample fire retardance and also eliminates the possibility of reducing the strength of the wood. In the case of interior trim there is no requirement for strength, which in the case of scaffolding and shoring is all important. Cost is another factor that cannot be overlooked and saturation treatments are expensive since they require a much longer treating cycle and also more chemicals.

III. TENTATIVE REVISION OF STANDARD

The Standard Specifications for Fire Tests of Building Construction and Materials (C 19–33; ASA A2-1934)[3] have served since 1933 with usefulness and satisfaction. Recently attention has been called to certain provisions in the application of which there has been a lack of uniformity. At a meeting of Subcommittee I (G. N. Thompson, chairman) it was agreed to recommend changes in the specifications that would help to minimize inconsistencies in testing procedure. Numerous other changes, chiefly of an editorial nature, were also recommended and it was decided to give further attention to several questions that have been raised. Among these are the effects of hose stream applications on structural members, and methods of determining transmitted temperatures.

These amendments will have the effect of simplifying the testing procedures, specifying them more definitely, and of giving a greater latitude in the size of test constructions. Their adoption will not invalidate results obtained in accordance with the present specifications, the main provisions of which have been found by experience to be well considered and well adapted for the ends in view.

While some modifications are proposed in the hose stream test requirements, a fuller study of the significance of this test as a criterion of the performance of building constructions during fires and extinguishing operations is to be made.

As a result of the discussion at the subcommittee meeting and further consideration by Committee C-5 at its March meeting, on the recommendation of Subcommittee I the following revisions of Standard Specifications C 19 are presented for publication as tentative:

Title and Introduction.—Change the title of Specifications C 19 by substituting the words "Methods of" for "Specifications for," making the title read "Standard Methods of Fire Tests of Building Construction and Materials"; also in the second and third paragraphs of the Introduction change "specifications" to read "methods."

It has been found that in certain constructions the increase in transmitted heat after the furnace has been shut off has been sufficient to cause the ignition of combustible material. It is accordingly recommended that the last paragraph of the introductory text be changed to read as follows by the addition of the italicized words:

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[3] 1939 Book of A.S.T.M. Standards, Part II, p. 152.

> Classifications of assemblies involving combustibles in such kind or quantity or so contained as to burn freely during the exposure to the test fire, or continue flaming *or ignite* after the furnace fire is shut off, shall be designated by the term "combustible" after the period assigned.

Section 3.—Experience has shown that for the types of construction to which these specifications apply at least five thermocouples are required for satisfactory results. Base metal thermocouples now generally used for furnace temperature measurements, have been found to function properly in black iron pipe. It has recently developed that no uniformity of practice exists at present with respect to the location of the thermocouples in the furnace. It is recommended that Paragraph (*a*) be changed to read as follows by the addition of the italicized words and the omission of those in brackets:

> 3. (*a*) The temperature fixed by the curve shall be deemed to be the average temperature obtained from the readings of [several thermocouples] not less than [three] *five thermocouples* symmetrically disposed and distributed to show the temperature near all parts of the sample, the thermocouples being enclosed in sealed porcelain tubes $\frac{3}{4}$ in. in outside diameter and $\frac{1}{8}$ in. in wall thickness, *or as an alternative in the case of base metal thermocouples, enclosed in sealed standard weight $\frac{1}{2}$-in. black wrought steel or black wrought iron pipe.* The exposed length of the [porcelain] *pyrometer* tube and couple in the furnace chamber shall be not less than 12 in. Other types of protecting tubes or pyrometers may be used that under test conditions give the same indications as the above standard within the limit of accuracy that applies for furnace-temperature measurements. [For greater difference of design and size, the time-temperature curve followed shall be modified to give an exposure equal to that obtained by using the standard pyrometer and curve above described.] *For floors and columns, the junction of the thermocouples shall be placed 12 in. away from the exposed face of the sample at the beginning of the test, and during the test, shall not touch the sample as a result of its deflection. In the case of walls and partitions, the thermocouples shall be placed 6 in. away from the exposed face of the sample at the beginning of the test, and shall not touch the sample during the test, in the event of deflection.*

Since the abandonment of wood as fuel with a resultant better control of temperatures, a lower permissible variation is not only possible but also highly desirable, and it is therefore recommended that Paragraph (*c*) be changed to read as follows by the addition of the italicized figures and the omission of those in brackets:

> (*c*) The accuracy of the furnace control shall be such that the area under the time-temperature curve, obtained by averaging the results from the pyrometer readings, is within [15] *10* per cent of the corresponding area under the standard time-temperature curve shown in Fig. 1 for fire tests of 1 hr. or less duration, within [10] *7.5* per cent for those over 1 hr. and not more than 2 hr., and within 5 per cent for tests exceeding 2 hr. in duration.

Section 4 (a).—Since the more general terms "protecting" and "insulating" seem preferable to the specific designation of the materials, it is recommended that the fifth sentence be changed to read as follows by the addition of the italicized words and the omission of those in brackets:

> The outside diameter of protecting *or insulating* tubes [of glass, clay, or porcelain,] and of thermometer stems shall be not more than $\frac{5}{16}$ in.

Section 7.—It happens occasionally that a construction, of which the fire-resistance rating is desired, does not, as actually installed, have as large dimensions as the minimum dimensions prescribed herein for the samples. It is believed that in such a case the possible risk of failure on a larger sample than actually used in practice should not be inflicted. It is accordingly recommended that the present Section 7 be designated as Paragraph (*a*) and that a new Paragraph (*b*) be added to read as follows:

> (*b*) The size and dimensions of the test samples specified herein are intended to apply for rating constructions of dimensions within the usual general range employed in buildings. If the conditions of use limit the construction to smaller dimensions, a proportionate reduction may be made in the dimensions of the samples for a test qualifying them for such restricted use.

Section 9 (*b*).—In order to make clear that the hose stream test is not required on constructions having ratings over ½ hr. but not of 1 hr., it is recommended that this paragraph be changed to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*b*) Exemption.—The hose stream test shall not be required in the case of constructions having a resistance period, indicated in the fire endurance test, of [30 min. or] less *than 1 hr.*

Table I.—Except for the third figure, 45, in the column headed "Water Pressure" the two parts of the present table are alike so far as water pressure and duration of application are concerned. It was agreed that this single figure could be changed from 45 to 30 without impairment of the test requirements. The words "if desired" are being added after the last item as the hose stream test in that case is optional. It is accordingly recommended that Table I be changed from its present form to read as follows:

TABLE I.

Resistance Period	Water Pressure at Base of Nozzle, psi.	Duration of Application, min. per 100 sq. ft. exposed area
8 hr. and over	45	6
4 hr. and over if less than 8 hr.	45	5
2 hr and over if less than 4 hr.	30	2½
1 hr. and over if less than 2 hr.	30	1½
Less than 1 hr. (if desired)	30	1

Sections 13 and 15.—Since in most cases fire-stopping is not needed, and in those cases where it is needed it must be made a part of the test sample (see Section 7), it is recommended that Paragraphs (*c*) in these two sections which read as follows be omitted and that the present Paragraphs (*d*) be designated Paragraphs (*c*):

> (*c*) The fire-stopping, if any, shall have functioned to prevent passage of fire for a period equal to that for which classification is desired.

Under the center heading reading "Tests of Floors and Roofs" following Section 18, change the parenthetical statement to read as follows by the omission of the words in brackets, as this part seems to be unnecessary:

> The following contemplates application of fire exposure to the under side of constructions [and omission from the upper surface of all units which are not essential to the constructions. Specifications and test procedure with fire applied to the upper side have not been developed].

The changes being recommended in the following sections are for the purpose of making the sections more truly expressive and removing any doubt as to their application.

Change the center heading immediately preceding Section 22 to read as follows by the addition of the italicized words and the omission of those in brackets:

> Tests of [Finish] *Protection* for Combustible Framing, or *for Combustible* Facings *on the Unexposed Side* of Walls, Partitions, and [Ceilings] *Floors.*

Sections 22 and 23.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 22. The test panel shall be a wall, partition, or floor.
>
> (*a*) Test panels [carrying] *having* interior wall and partition [finish] *protection* shall be finished on both faces with the [finish] *protection*

which is the subject of the test, [except that with the advice or consent of the testing body this provision may be waived with respect to panels of solid construction] *or with an interior wall finish on the unexposed side judged by the testing authority to be suitable for the purposes of the test.*

(*b*) Test panels [carrying] *having* exterior wall [finish] *protection* on the exposed face shall be finished on the unexposed face with an interior wall finish judged by the testing body to be suitable for purposes of the test [except that with the advice or consent of the testing body this provision may be waived with respect to panels of solid construction].

(*c*) Test panels [carrying] *having a protective* ceiling [finish] *or slab* shall be finished on the upper face with a flooring judged by the testing body to be suitable for purposes of the test [except that with the advice or consent of the testing body this provision may be waived with respect to panels of solid construction].

23. The area exposed to fire shall be, for tests of wall and partition [finish] *protection*, not less than 100 sq. ft. with neither dimension less than 9 ft.; for tests of [ceiling finish] *floor protection*, not less than 180 sq. ft. with neither dimension less than 12 ft.

Section 24.—Change Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

(*a*) The [finish] *protection* shall have withstood the fire endurance test, without [passage of flame or of gases hot enough to ignite] *ignition of* the materials protected, for a period equal to that for which classification is desired.

Omit Paragraph (*b*) which reads as follows:

(*b*) The finish shall have withstood the fire and hose stream test as prescribed respectively for floors, walls, and partitions as specified in Section 9, without passage of flame, of gases hot enough to ignite the materials protected, or of the hose stream.

Reletter Paragraph (*c*) as (*b*) and change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*b*) Transmission of heat through the [finish] *protection* during the fire endurance test shall not have been such as to raise the temperatures at its contact with the *protected* structural members *or facings* of the test panel [or elsewhere on its unexposed surface] more than 250 F. (139 C.) above the initial temperatures at these points, *except that for members closely embedded on three sides in masonry, concrete or other incombustible materials the permissible temperature rise may be 325 F. (181 C.).*

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballot Marked "Not Voting"
I New Tentative Standard			
Fire Tests of Door Assemblies.	18	0	2
II. Revision of Tentative Standard			
Spec. for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 - 37 T)........	18	1	1
III. Tentative Revision of Standard			
Spec. for Fire Tests of Building Construction and Materials (C 19 - 33; ASA A2-1934)........	18	1	1

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 30 members; 20 members returned their ballots, with the results shown in Table II.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 30 members; 26 members returned their ballots, of whom 25 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. P. MILLER,
Chairman.

H. M. ROBINSON,
Secretary.

REPORT OF COMMITTEE C-7
ON
LIME

Committee C-7 on Lime has held but one meeting during the year: on June 28, 1939, in Atlantic City, N. J.

Three resignations have occurred, two of which have resulted in reducing the total membership of the committee to 48, classified as 24 producers, 6 consumers, and 18 general interest members.

Subsequent to the 1939 annual meeting, Committee C-7 presented to the Society through Committee E-10 on Standards a recommendation for the withdrawal of the Tentative Specifications for Quicklime for Structural Purposes (C 5 – 34 T), and for Hydrated Lime for Structural Purposes (C 6 – 34 T). This recommendation was made in view of the previous action of Committee C-7 in deleting the soundness test as described in Sections 4 and 5 of the Tentative Methods of Physical Test for Limestone, Quicklime, and Hydrated Lime (C 110 – 34 T), to which reference was made in the tentative structural lime specifications. The existence of adequate Standard Specifications for Quicklime for Structural Purposes (C 5 – 26), and for Hydrated Lime for Structural Purposes (C 6 – 31) was a further reason for suggesting that the obsolete tentative standards be withdrawn. This recommendation was accepted[1] by Committee E-10 at its meeting on August 24, 1939.

I. Tentative Revision of Standard

The committee approved the proposed revision of the Standard Specifications for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 – 24)[2] as submitted by Subcommittee VII on Specifications of Committee C-8 on Refractories[3] and recommends that this revision, given below, be published as tentative:

Section 1.—Change the note following this section from its present form to read as follows:

Note.—In the manufacture of silica brick, silica in the form of massive quartzite or quartz conglomerate is ground until the particles are less than ¼ in. in size. Lime either in the form of slaked lime or hydrated lime is then added in quantities varying from 1.5 to 3.0 per cent CaO and the shapes are molded and dried. They are then burned in down-draft or tunnel kilns until most of the quartzite has been converted into tridymite or cristobalite.

Section 3.—In the table of chemical composition change the requirement for calcium oxide from "92.0 per cent" to read "90 per cent," and increase the permissible magnesium oxide content from "3.0 per cent" to read "4.5 per cent."

This recommendation has been submitted to letter ballot of the committee which consists of 47 members; 30 members returned their ballots, of whom 18 have voted affirmatively, 0 negatively, and 12 members marked their ballots "not voting."

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee C-7 reported results of the letter ballot vote as follows: Of a total membership of 50, 36 members returned their ballots, of whom 32 voted affirmatively, 0 negatively, and 4 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 34.

[3] See p. 265.

II. TENTATIVE STANDARDS CONTINUED WITHOUT REVISION

In the absence of specific consideration during the year, the committee recommends continuing without revision the Tentative Methods of Physical Testing of Quicklime and Hydrated Lime (C 110 – 38 T), and the Tentative Specifications for Hydraulic Hydrated Lime for Structural Purposes (C 141 – 38 T).

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Nathan C. Rockwood.

First Vice-Chairman, Walter C. Voss.

Second Vice-Chairman, William E. Carson.

Secretary, W. Vernon Brumbaugh.

This report has been submitted to letter ballot of the committee which consists of 47 members; 43 members returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. WITHROW,
Chairman.

W. V. BRUMBAUGH,
Acting Secretary.

REPORT OF COMMITTEE C-8

ON

REFRACTORIES

Committee C-8 on Refractories held two meetings during the year: one on September 7, 1939, at Conneaut Lake, Pa., in conjunction with the Refractories Division of the American Ceramic Society, and the other on March 7, 1940, at Detroit, Mich., in conjunction with the spring group meetings of the A.S.T.M.

The committee has been very active during the year, and action has been taken on many standards. Considerable work was done, particularly by the Editorial Subcommittee, in preparing the standards of the committee for printing in the new double column format in the 1939 Book of A.S.T.M. Standards. Changing from single- to double-column pages necessitated many editorial changes in both text and tables. In order to include as many standards on refractories as possible in the 1939 Book of Standards, the committee submitted the following recommendations, resulting from the September meeting, to the Society through Committee E-10 on Standards:

Tentative Methods of Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Products (C 20 - 39 T), to replace immediately the Standard Method of Test for Porosity and Permanent Volume Changes in Refractory Materials (C 20 - 33),

Tentative Definitions of Terms Relating to Refractories (C 71 - 39 T), including definitions of insulating fire brick, insulating block, calcining, and burning (firing), to replace the definitions of insulating brick, hard-, light-, and medium-burned refractory ware appearing in Tentative Definitions C 71 - 37 T, and

Tentative revision of Standard Symbols for Heat Transmission (C 108 - 39), involving the addition of 5 symbols, proposed by the American Standards Association, referring to specific heat per unit weight, density weight per unit volume, thermal diffusivity, Stefan-Boltzmann constant, and total emissivity.

These recommendations were accepted[1] by Committee E-10 on December 4, 1939, and the new tentative standards and tentative revision appear in the 1939 Book of A.S.T.M. Standards, Part II.

I. New Tentative Standards

Tentative Specifications for Refractories:

The Standard Specifications for Fireclay Brick for Malleable Furnaces with Removable Bungs and for Annealing Ovens (C 63 – 39), for Stationary Boiler Service (C 64 – 39), and for Refractories for Construction of Incinerators (C 106 – 39) have not been changed appreciably for a number of years. As a result of presenting the new classification of fireclay brick as a tentative revision of the Standard Definitions of Fireclay and Alumina-Diaspore Refractories (C 27 – 39), discussed in detail later in this report, the committee decided to incorporate the new classification in the three specifications mentioned above. Accordingly, new tentative specifications have been prepared to replace the present Standard Specifications C 63, C 64, and C 106. In so doing, the presentation of the subject matter has been improved and Specifications C 64 have been separated into two specifications, one for heavy duty service and the other for moderate duty service. It is accordingly recommended that the following four specifications be accepted for publication as tentative to replace immediately the corresponding standard specifications:

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee C-8 reported the following results of the letter ballot vote of a total of 23 ballots returned from a committee membership of 33: C 20 – 39 T and C 71 – 39 T, affirmative 23, negative 0; C 108 – 39, affirmative 21, negative 0, ballots marked "not voting" 2.

Tentative Specifications for:[2]

Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens, to replace Standard Specifications C 63 - 39,

Refractories for Heavy Duty Stationary Boiler Service, to replace in part Standard Specifications C 64 - 39,

Refractories for Moderate Duty Stationary Boiler Service, to replace in part Standard Specifications C 64 - 39, and

Refractories for Incinerators, to replace Standard Specifications C 106 - 39.

Tentative Method of Test for Warpage of Refractory Brick and Tile:[3]

The Tentative Methods of Test for Size, Warpage, and Bulk Specific Gravity of Refractory Brick (C 134 – 38 T) were revised last year to cover only a test for size and bulk density of refractory brick (C 134 – 39 T), the method for measuring warpage to be the subject of a separate procedure. An improved method of determining warpage has now been developed and the committee accordingly recommends that the Tentative Method of Test for Warpage of Refractory Brick and Tile be accepted for publication as tentative.[3]

Tentative Classification of Insulating Block and Insulating Fire Brick:

A classification of insulating block and insulating fire brick, based on shrinkage as determined by heat treatment at specified temperatures, and by bulk density, has been prepared. It is recommended that this classification be accepted for publication as tentative.[4]

II. REVISION OF TENTATIVE STANDARD

Tentative Definitions of Terms Relating to Refractories (C 71 – 39 T):[5]

The need for acceptable definitions of the terms abrasion, corrosion, erosion, and slagging became particularly apparent in the preparation of industrial surveys which involved the use of these words. The following precise and practical definitions are accordingly presented with the recommendation that they be added to Tentative Definitions C 71 – 39 T:

Abrasion.—Wearing away of refractory surfaces by the scouring action of moving solids.

Corrosion.—Destruction of refractory surfaces by the chemical action of external agencies.

Erosion.—Wearing away of refractory surfaces by the washing action of moving liquids, such as molten metals and slags.

Slagging.—Destructive chemical reaction between refractories and external agencies at high temperatures, resulting in the formation of a liquid.

III. TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Ground Fire Clay (C 105 – 36):[5]

The committee feels that certain changes are desirable in these specifications. The screen-size requirements need to be made more rigid, the moderate heat duty type of fire clay is omitted from the new classification of fireclay brick and therefore should be deleted from these specifications, and there is need for a super duty class of clay for use with super duty fireclay brick. Also a method of sampling shipments of ground clay in bulk and containers is necessary if suitable laboratory samples are to be obtained for test purposes. Accordingly, the committee recommends that the following revisions of Standard Specifications C 105 be accepted for publication as tentative:

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[2] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 199, 202, 206, 208, respectively.

[3] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 218.

[4] This classification was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 221.

[5] 1939 Book of A.S.T.M. Standards, Part II, pp. 970, 159.

Section 4.—Change from its present form: namely,

> 4. (*a*) *Commercial Grade.*—The commercial grade shall be of such a particle size that 90 per cent of the material passes a No. 20 A.S.T.M. sieve (equivalent to mesh No. 20 of the Tyler standard series).
>
> (*b*) *Fine Grade.*—The fine grade shall be of such a particle size that 90 per cent of the material passes a No. 30 A.S.T.M. sieve (equivalent to mesh No. 28 of the Tyler standard series).

to read as follows:

> 4. (*a*) *Commercial Grade.*—Not more than 5 per cent shall be retained on a No. 20 A.S.T.M. sieve (equivalent to mesh No. 20 of the Tyler standard series), and not more than 0.5 per cent shall be retained on a No. 16 A.S.T.M. sieve (equivalent to mesh No. 14 of the Tyler standard series).
>
> (*b*) *Fine Grade.*—Not more than 5 per cent shall be retained on a No. 30 A.S.T.M. sieve (equivalent to mesh No. 28 of the Tyler standard series), or more than 0.5 per cent on a No. 20 A.S.T.M. sieve (equivalent to mesh No. 20 of the Tyler standard series).

Section 5.—At the beginning of the table of pyrometric cone equivalent requirements add a new class of fire clay to read: "Super duty . . . not below No. 31."

Omit the requirement which read: "Moderate heat duty . . . not below No. 20."

Sampling.—Add a method of sampling as Section 7 to read as follows:

> 7. *Sampling:* (*a*) *Containers.*—When shipped in containers, a sample shall be taken as follows: If the shipment consists of 100 containers or fraction thereof, the contents of one container, selected at random, shall be dumped and quartered to provide a 5-lb. sample for the laboratory. If the shipment consists of more than 100 containers, the number of containers to be sampled shall be as follows:
>
> | Number of Containers in Shipment | Number of Containers Selected for Sampling |
> |---|---|
> | Over 100 but less than 300 | 2 |
> | 300 but less than 600 | 3 |
> | 600 but less than 1000 | 4 |
>
> In these cases, the several 5-lb. samples resulting from quartering shall be combined and either quartered or riffled to provide a 5-lb. sample for the laboratory.
>
> (*b*) *Bulk.*—When shipped in bulk, the sample shall be so taken that it will represent an average of all parts of the shipment from top to bottom and shall not contain a disproportionate share of the top and bottom layers. For a 30-ton unit, no less than 10 samples of approximately 25 lb. each shall be taken for different parts of the car or truck by digging holes spaced at equal intervals. If the shipment is more than 30 tons, a sample proportionately larger shall be taken. The combined samples shall be thoroughly mixed and quartered or riffled to provide a 5-lb. sample for the laboratory.
>
> It shall be permissible to obtain the sample during unloading by taking a small scoopful of clay at regular intervals. One scoopful of clay, weighing approximately 1½ lb., shall be taken for each ton of material. In no case, however, shall the collected sample weigh less than 50 lb. The collected sample shall be well mixed and quartered or riffled to provide a 5-lb. sample for the laboratory.

Standard Methods of Chemical Analysis of Refractory Materials (C 18 – 39):[6]

Several years of cooperative study have produced a rapid and accurate method of analysis of chrome ores and chrome refractories which is being presented as a tentative revision of Sections 51 to 55 of Standard Methods C 18. The new method is being presented to the Society for publication as a Tentative Method of Chemical Analysis of Chrome Ore and Chrome Refractories[7] and is intended to replace, when adopted as standard, Sections 51 to 55 of Methods C 18.

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[6] 1939 Book of A.S.T.M. Standards, Part II, p. 173.

[7] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 211.

Standard Definitions of Fireclay and Alumina-Diaspore Refractories (C 27 – 39):[8]

In Definitions C 27 high heat duty fireclay brick are defined by their P.C.E. value, and a separate definition is given for siliceous fireclay brick. A new set of classifications, rather than definitions, is being presented by the committee to classify high and intermediate heat duty fireclay brick according to refractoriness as measured by P.C.E. *or* by means of their behavior in the load test. The P.C.E. requirement is intended to pertain to the more usual fireclay refractories, while the load-test requirement pertains specifically to the siliceous type of fireclay brick. For the low heat duty fireclay brick, the P.C.E. is adequate for both types. The load test has for years been used as a measure of refractoriness. As the classification of moderate heat duty fireclay brick is no longer in common use, the committee is recommending that it be omitted from the list. It is accordingly recommended that the following revision[9] of Definitions C 27 be accepted for publication as tentative, to replace the tentative revision presented for publication in 1938:

Sections 3 and 4.—Change the definitions of high heat duty fireclay brick and intermediate heat duty fireclay brick from their present form to read as follows:

> 3. *High Heat Duty Fireclay Brick* shall be required to conform to only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 31–32, *or* not more than 1.5 per cent deformation in the 1350 C. (2462 F.) load test.
>
> 4. *Intermediate Heat Duty Fireclay Brick* shall be required to conform to only *one* of the following requirements: Pyrometric cone equivalent not lower than cone No. 29, *or* not more than 3 per cent deformation in the 1350 C. (2462 F.) load test.

Section 5.—Omit the definition of moderate heat duty fireclay brick, renumbering the subsequent sections accordingly.

Section 6.—Renumber as Section 5 and change the definition of low heat duty fireclay brick from its present form to read as follows:

> 5. *Low Heat Duty Fireclay Brick* shall have a pyrometric cone equivalent not lower than cone No. 19.

IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD

Tentative Method of Test for True Specific Gravity of Burned Refractory Materials (C 135 – 38 T):[10]

This method has not been the subject of any constructive criticism since its publication as tentative in 1938. The committee accordingly recommends that it be approved for reference to letter ballot of the Society for adoption as standard.

V. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

The committee recommends that the revisions of the following two methods of test, published as tentative in 1939, referring to the standardization of furnace gas pressure during preheating, be approved for reference to letter ballot of the Society for adoption as standard:

Standard Methods of:

Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 - 36), and

Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 - 37).

VI. WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standard specifications since these are being replaced by new tentative specifications, as mentioned earlier in this report:

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[8] 1939 Book of A.S.T.M. Standards, Part II, p. 173.

[9] This revision has been incorporated in the revised Tentative Classification of Fireclay Refractories (C 27 – 40 T) which was accepted for publication as tentative by Committee E-10 on Standards, see Editorial Note, p. 266.

[10] 1939 Book of A.S.T.M. Standards, Part II, p. 968.

Standard Specifications for:[11]

Fireclay Brick for Malleable Furnaces with Removable Bungs and for Annealing Ovens (C 63 - 39),

Fireclay Brick for Stationary Boiler Service (C 64 - 39) and

Refractories for Construction of Incinerators (C 106 - 39).

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (C 63 - 40 T)	24	0	11
Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 - 40 T)	26	1	8
Spec. for Refractories for Moderate Duty Stationary Boiler Service	27	0	8
Spec. for Refractories for Incinerators (C 106 - 40 T)	26	0	9
Test for Warpage of Refractory Brick and Tile	34	1	0
Classification of Insulating Block and Insulating Fire Brick[a]	31	4	0
II. REVISION OF TENTATIVE STANDARD			
Definitions of Terms Relating to Refractories (C 71 - 39 T)	34	1	0
III. TENTATIVE REVISIONS OF STANDARDS			
Spec. for Ground Fire Clay (C 105 - 36)	31	1	3
Chemical Analysis of Refractory Materials (C 18 - 39)	29	0	6
Definitions of Fireclay and Alumina-Diaspore Refractories (C 27 - 39)	29	2	4
IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Test for True Specific Gravity of Burned Refractory Materials (C 135 - 38 T)	32	0	3
V. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 - 36)	33	0	2
Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 - 37)	33	0	2
VI. WITHDRAWAL OF STANDARDS			
Spec. for Fireclay Brick for Malleable Furnaces with Removable Bungs and for Annealing Ovens (C 63 - 39)	24	0	11
Spec. for Fireclay Brick for Stationary Boiler Service (C 64 - 39)	26	1	8
Spec. for Refractories for Construction of Incinerators (C 106 - 39)	26	0	9
VII. EDITORIAL CHANGE			
Test for Size and Bulk Density of Refractory Brick (C 134 - 39 T)	33	1	1

[a] The classified vote on the proposed Tentative Classification of Insulating Block and Insulating Fire Brick was as follows: Affirmative: 15 producers, 9 consumers, 7 general interests; negative: 2 producers, 1 consumer, 1 general interest.

VII. EDITORIAL CHANGE

Tentative Methods of Test for Size and Bulk Density of Refractory Brick (C 134 - 39 T):[12]

In connection with this method a study has shown that it is useless to make measurements of over 9 in. to an accuracy greater than $\frac{1}{32}$ in. Since the present method specifies $\frac{1}{64}$ in. it is accordingly recommended that an editorial change be made in the requirements set forth in Section 5 (*a*) by adding the following to the first sentence: "when the dimensions are 9 in. or under, and to the nearest $\frac{1}{20}$ or $\frac{1}{30}$ in. when the dimensions are over 9 in."

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 35 members; all the members returned their ballots with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (L. C. Hewitt, chairman) is preparing industrial surveys on refractory materials in the zinc and cement industries. Arrangements have been made to secure the assistance of an authority in the field to assist in preparing a report on a boiler service survey. It is likely that a year will be required to complete these projects.

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[11] 1939 Book of A.S.T.M. Standards, Part II, pp. 161, 165, 170.

[12] *Ibid.*, p. 966.

Subcommittee II on Research (R. S. Bradley, chairman) is continuing to work in close cooperation with Subcommittee III on Tests. The group is also keeping in touch with the work at the National Bureau of Standards on low and high temperature spalling studies.

Subcommittee III on Tests (F. A. Harvey, chairman) is composed of the chairmen of the seven sections mentioned below. The activities of this subcommittee are brought out in the foregoing recommendations on standards, and in the following discussion of the activities of the various sections.

Section A on Load in 1939 proposed a new equipment for use in testing refractories under load at high temperatures. This was accepted by the Society with the result that there are now three types of furnaces for use in conducting the load test (A.S.T.M. Methods C 16 – 39 T). Having completed this work, the activities of the section have been only nominal during the past year.

Section B on Spalling (R. E. Birch, chairman) is recommending the adoption as standard of the tentative revision of the panel tests (C 107 – 36 and C 122 – 37), referred to earlier in this report.

The section, in cooperation with the Refractories Fellowship at Mellon Institute, has been studying the subject of a revised design for the back-up insulation, used during the preheating period, of the panels in the panel-spalling test. The section believes that the study is nearing completion and that it will have a proposal to offer for consideration at the fall meeting of Committee C-8.

Section C on Temperature (R. B. Sosman, chairman) has prepared a recommendation for use by the manufacturers of pyrometric cones in order to supply the trade with uniform test cones Nos. 19 to 42 (small cones) for use in determining P.C.E. according to Standard Method C 24 – 35. This recommendation, given below, may eventually be presented to the Society for adoption as standard:

> 1. *Shape of the Test Cone.*—A truncated trigonal pyramid with its base at a small angle to the trigonal axis.
>
> 2. *Dimensions.*—Over-all height (between planes perpendicular to the trigonal axis) $1\frac{1}{8}$ in. Length of side of the truncated tip $\frac{1}{32}$ in. Length of the two longer sides of the base $\frac{9}{32}$ in. Base at an external angle of 82 deg. to one face and perpendicular to the symmetry plane through the opposite edge of the pyramid.
>
> 3. *Depth of immersion in the supporting plaque*, which is to be $\frac{1}{8}$ in., is to be marked by a line, parallel to the base, on one or more faces.
>
> 4. *Permissible variations in dimensions* are 3 per cent in height, 5 per cent in width of base. (Subject to change after further experience with pressed cones.)
>
> 5. *The number of the cone* is to be impressed on one of the two faces which are not at 82 deg. to the base.

Section E on Analysis (L. J. Trostel, chairman), as a result of several years of cooperative study, has prepared an improved method of analyzing chrome ore and chrome refractories, which is being recommended for publication as a tentative revision of Methods C 18 – 39. The method incorporates procedures which are comparatively rapid, but which are accurate.

Section F on Tests on Refractory Insulation (W. R. Kerr, chairman) has prepared a classification of insulating refractories based on permanent linear change data and weight per cubic foot, which is being presented for publication as tentative. The classification covers five groups of insulating refractories.

This section is also working in close cooperation with Committee C-16 on Thermal Insulating Materials for the purpose of eventually developing a transverse-strength test for insulating block.

An effort is being made to obtain the cooperation of several laboratories to run simulative service panel tests on insulating fire brick to compare the results with the present linear change test on reheating now included in Methods C 93 – 39 T.

Section G on Porosity and Permanent Volume Change (C. E. Fulton, chairman) at the September meeting presented new Tentative Methods of Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Products (C 20 – 39 T) which, as mentioned earlier in this report, were accepted by Committee E-10 on Standards for publication.

Subcommittee IV on Heat Transfer (R. H. Heilman, chairman) is working on the determination of the thermal conductivity of block insulation. Samples of suitable material were sent to a number of laboratories to learn something of the order of results which may be obtained with different equipment and by different operators. These tests have been completed. At a mean temperature of 1300 F., the maximum variation of all the test results reported was approximately 22 per cent, or a plus or minus variation of 11 per cent. At lower mean temperatures, the agreement was not so good.

The National Bureau of Standards reports that after many difficulties it has been getting rather consistent results with the flow calorimeter. Two sets of alundum blocks have been calibrated up to about 1000 F. to use as flow calorimeters or heat flow meters. The Bureau is now proceeding to calibrate these at higher temperatures by the stepping-up process. The section plans to meet with M. S. Van Dusen in the near future and discuss the progress of the work he is conducting at the Bureau.

Subcommittee V on Precision and Tolerance (R. A. Heindl, chairman) has developed a clay sampling procedure. This has been submitted to Committee C-8 through Subcommittee VII on Specifications, and is being recommended at this time for publication as a tentative revision of the Standard Specifications for Ground Fire Clay (C 105 – 36).

Subcommittee V is also revising the Recommended Methods for Interpretation of Refractory Test Data (1937).[13] This work may take another year before completion.

Section A on Retest Data (L. J. Trostel, chairman) is accumulating data that will be useful in working out retest requirements.

Subcommittee VI on Nomenclature (M. E. Holmes, chairman) presented to Committee C-8 at the September meeting new definitions for insulating fire brick, insulating block, calcining, and burning (firing) (C 71 – 39 T) which, as mentioned earlier in this report, were accepted by Committee E-10 on Standards for publication. A revision of the Standard Symbols for Heat Transmission (C 108 – 39) was also accepted for publication as tentative, which added 5 new symbols for heat transmission. This revision was proposed by Subcommittee VI on the recommendation of Subcommittee IV.

Definitions for abrasion, corrosion, erosion, and slagging have been prepared and are now being presented to the Society for publication as a revision of Tentative Definitions C 71 – 39 T.

Subcommittee VI has also presented a new classification of high heat duty fireclay brick and low heat duty fireclay brick, which is being recommended for publication as a tentative revision of Definitions C 27 – 39.

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[13] "Manual of A.S.T.M. Standards on Refractory Materials," p. 108 (1937), issued as a separate publication.

Subcommittee VI also presented to Committee C-8, on the recommendation of Section F of Subcommittee III, the previously discussed classification of insulating fire brick.

This active subcommittee is now preparing additional necessary definitions.

Subcommittee VII on Specifications (G. A. Bole, chairman), in addition to preparing the new tentative specifications, discussed earlier in this report, for refractories for boiler service, for malleable iron furnaces and annealing ovens, and for incinerators, and the revision of the ground fire clay specifications (C 105 – 36), has proposed a revision of the Standard Specifications for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 – 24)[14] which is under the jurisdiction of Committee C-7 on Lime. Committee C-8 is recommending to Committee C-7 that the specifications be changed as follows:[15]

The following is proposed to replace the present Note in Section 1:

NOTE.—In the manufacture of silica brick, silica in the form of massive quartzite or quartz conglomerate is ground until the particles are less than ¼ in. in size. Lime either in the form of slaked lime or hydrated lime is then added in quantities varying from 1.5 to 3.0 per cent CaO and the shapes are molded and dried. They are then burned in down-draft or tunnel kilns until most of the quartzite has been converted into tridymite or cristobalite.

In addition, in the table of chemical requirements in Section 3 it is proposed to change the requirement for calcium oxide from "92.0 per cent" to read "90 per cent," and to increase the permissible magnesium oxide content from "3.0 per cent" to read "4.5 per cent."

Subcommittee VIII, Editorial (L. J. Trostel, chairman) has been active during the year and has cooperated with the Society in changing the refractories standards from single to double column format for publication in the 1939 Book of A.S.T.M. Standards.

A new edition of the Refractories Manual is planned for the fall of 1940, and this will incorporate the latest revisions of the C-8 literature. It is possible that certain actions to be taken at the fall meeting of the committee can be incorporated in the manual. The industrial surveys pertaining to refractory service in lime burning and in plate glass and window glass furnaces will be included, and it is possible that the one pertaining to boiler furnace refractories will be ready by that time. There is a distinct need for a new edition of the manual, which is finding an important place in the literature on refractories.

At the March, 1940, meeting the following were elected as officers of Committee C-8 for the ensuing 2-year period:

Chairman, J. D. Sullivan.
Vice-Chairman, L. J. Trostel.
Secretary, S. M. Phelps.

This report has been submitted to letter ballot of the committee which consists of 35 members; 34 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

J. D. SULLIVAN,
Chairman.
E. H. VAN SCHOICK,
Vice-Chairman.

S. M. PHELPS
Secretary.

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[14] 1939 Book of A.S.T.M. Standards, Part II, p. 34.

[15] See Report of Committee C-7 on Lime, p. 256.

EDITORIAL NOTE

Subsequent to the annual meeting Committee C-8 presented to the Society through Committee E-10 on Standards the following recommendations:

Withdrawal of Standard:

Definitions of Fireclay and Alumina-Diaspore Refractories (C 27 - 39) and publication as tentative of the revised Classification of Fireclay Refractories (C 27 - 40 T).

Tentative Revisions of Standard:

Method of Test for Pyrometric Cone Equivalent of Refractory Materials (C 24 - 35),

Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 - 36),

Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 - 40), and

Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 - 40).

These recommendations were accepted by Committee E-10 on November 5, 1940, and the revised tentative definitions and tentative revisions appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 219, 342, and 343.

REPORT OF COMMITTEE C-9
ON
CONCRETE AND CONCRETE AGGREGATES

Committee C-9 on Concrete and Concrete Aggregates held three meetings during the year: two in Washington, D. C., on December 9, 1939, and on March 20, 1940, and one during the annual meeting of the Society in Atlantic City, N. J.

The first Sanford E. Thompson Award is to be made this year, T. F. Willis and M. E. DeReus being the recipients for their paper on "Thermal Volume Change and Elasticity of Aggregates and Their Effect on Concrete," which was presented at the 1939 annual meeting of the Society.[1] This award was established by Committee C-9 as an annual token of recognition to the author or authors of a paper of outstanding merit on concrete or concrete aggregates presented at an annual meeting of the Society. The purposes of the award, which is named in honor of the first chairman of the committee, are to stimulate research, encourage the presentation of results of investigations that extend the knowledge of concrete and concrete aggregates, and to recognize meritorious efforts.

The election of officers for the ensuing term of two years has resulted as follows:

Chairman, F. H. Jackson.
Vice-Chairman, F. E. Richart.
Secretary, R. R. Litehiser.
Chairman of Finance Committee, H. J. Love.

Members-at-Large of Advisory Committee:

H. F. Clemmer	F. R. McMillan
R. W. Crum	J. C. Pearson
H. J. Gilkey	C. H. Scholer
A. T. Goldbeck	Stanton Walker

Dr. A. N. Talbot was again unanimously elected honorary chairman in appreciation of his many years of service to the committee.

I. New Tentative Standards

The committee recommends that the two methods of test listed below be accepted for publication as tentative. For some time there has been a definite need for both these methods and they are considered an outstanding contribution to the accomplishments of the committee.

Tentative Methods of:[2]

Test for Efficiency of Materials for Curing Concrete, proposed by Subcommittee XIII (W. F. Kellermann, chairman), and

Test for Volume Change of Cement Mortar and Concrete, proposed by Subcommittee XVI (L. W. Teller, chairman).

II. Revisions of Standards

The committee recommends that the following revision in the Standard Specifications for Concrete Aggregates (C 33 – 39)[3] be approved for immediate adoption:

Section 11 (*k*).—Omit this section which reads as follows, as the method referred to has been discontinued:

> (*k*) *Soft Fragments.*—Method of Test for Quantity of Soft Pebbles in Gravel, Standard Method T-8 of The American Association of State Highway Officials.

The committee recommends that the following revision of the Standard

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 919 (1939).

[2] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 264 and 266.

[3] 1939 Book of A.S.T.M. Standards, Part II, p. 281.

Method of Test for Compressive Strength of Concrete (C 39 – 39)[4] be accepted for publication as tentative, as proposed by Subcommittee VII (A. T. Goldbeck, chairman):

Section 15.—Change from its present form: namely,

> 15. Concrete test specimens shall be removed from the molds 20 to 48 hr. after molding, marked, weighed, and stored in damp sand, under damp cloths, or in a moist chamber until the date of test. The temperature of the curing room should not fall outside the range of 18 to 24 C. (65 to 75 F.).

to read as follows:

> 15. The concrete test specimens shall be removed from the molds not earlier than 20 nor later than 48 hr. after molding and stored in a moist condition at a temperature within the range of 65 to 75 F. (18 to 24 C.) until test. Moist condition shall be considered as that which maintains free moisture on the surface of the specimens at all times. Specimens shall not be exposed to a stream of running water; if stored in water, it shall be a saturated lime solution.
>
> NOTE.—Attention is directed to the fact that the temperature within the damp sand and under wet burlap or similar materials will always be lower than the temperature in the surrounding atmosphere if evaporation takes place.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 70 members, with the results shown in Table I.

This report has been submitted to letter ballot of the committee which consists of 70 members; 56 members returned their ballots, all of whom have voted affirmatively.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Efficiency of Materials for Curing Concrete...	46	3	7
Test for Volume Change of Cement Mortar and Concrete....................	48	2	6
II. REVISIONS OF STANDARDS			
Spec. for Concrete Aggregates (C 33 – 39), immediate adoption..........................	52	1	2
Test for Compressive Strength of Concrete (C 39 – 39)......	51	1	4

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

R. R. LITEHISER,
Secretary.

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[4] 1939 Book of A.S.T.M. Standards, Part II, p. 328.

REPORT OF COMMITTEE C-11

ON

GYPSUM

Committee C-11 on Gypsum has held one meeting during the past year, at the National Bureau of Standards in Washington, D. C., on March 19, 1940.

The present officers of the committee were re-elected for the ensuing term of two years.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Gypsum Plasters (Miller Porter, chairman).—Additional tests have been conducted during the past year by members of this subcommittee to determine the difference in strength of Keene's cement briquets poured at consistencies of 20-mm. and 30-mm. penetration. These tests completed the work on Keene's cement and the revisions of the Standard Specifications for Keene's Cement (C 61 – 30) published as tentative in 1938 are now being recommended for adoption as standard.

Subcommittee III on Structural Gypsum Products (L. S. Wells, chairman).—This subcommittee is now reviewing the Federal Specification for Gypsum Partition Tile (SS – T – 316) with a view to revising the A.S.T.M. Specifications for Gypsum Partition Tile or Block (C 52 – 33) to conform to the Federal Specification.

Subcommittee IV on Testing Methods (C. K. Roos, chairman).—Several members of this subcommittee have checked the elutriation method of determining the wood fiber content in wood-fibered gypsum plaster, published in 1939 as a tentative revision of the Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 39). The method has been found reasonably accurate and simple and it is therefore recommended that it be adopted as standard.

It has been suggested that the ammonium acetate method for gypsum analysis be incorporated in Methods C 26 as an alternate method. Several members have conducted tests to compare the present gravimetric method with the ammonium acetate method. Additional tests will be made during the year by more members of this committee and it is possible that an alternate method will be suggested next year for publication as tentative.

I. REVISION OF TENTATIVE STANDARD

Tentative Definitions of Terms Relating to Gypsum (C 11 – 31 T).[1]—Perforated gypsum lath is designed to provide 1-hr. fire resistance when plastered with ½ in. of gypsum plaster. To obtain this fire resistance, the perforations must be not less than ¾ in. in diameter and must comprise not less than 2.75 per cent of the area of the lath. The committee accordingly presents for publication as tentative the following new definition to be added to Tentative Definitions C 11 – 31 T:

> *Perforated Gypsum Lath.*—A gypsum lath with perforations not less than ¾ in. in diameter, distributed over the face of the lath, and comprising not less than 2.75 per cent of the area of the lath.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 915.

II. REVISIONS OF STANDARDS

Standard Specifications for Gypsum Plasters (C 28 – 39; ASA A49.3–1939), Gypsum Molding Plaster (C 59 – 30; ASA A49.4–1933), Gypsum Pottery Plaster (C 60 – 30; ASA A49.5–1933), Keene's Cement (C 61 – 30), and Calcined Gypsum for Dental Plasters (C 72 – 30).[2]—In Section 22 (*b*) of Standard Specifications C 28 and in Section 9 (*b*) of Standard Specifications C 59, C 60, C 61, and C 72, it is required that the net weight of the package be shown on each package or tag. Inasmuch as most state laws require that the gross weight be shown, the committee recommends that in the indicated sections of these specifications the words "net weight of package" be revised to read "net and gross weights of package." The committee accordingly asks for the necessary nine-tenths vote at the annual meeting in order that this modification may be referred to letter ballot of the Society for immediate adoption.

Standard Specifications for Gypsum Lath (C 37 – 34)[3].—Gypsum lath is now manufactured in plain and perforated types, both of which are made to conform to Standard Specifications C 37. The committee recommends for immediate adoption the following revision in Standard Specifications C 37 and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that this change may be referred to letter ballot of the Society.

Section 1.—Change to read as follows by the addition of the italicized words:

> 1. These specifications cover *both plain and perforated* gypsum lath, gypsum plaster boards designed to be used as a base for the reception of gypsum plaster.

Standard Specifications for Gypsum (C 22 – 25; ASA A49.1–1933)[4].—The committee has long felt that the minimum requirement for $CaSO_4 \cdot 2H_2O$ content in Section 1 (*b*) of Specifications C 22 is too low and could very well be increased. It therefore presents for publication as tentative the revision given below.

In Section 7 (*b*) under the heading Packing and Marking, it is required that on the tag attached to each package there be listed among other things the form and size of material, and the chemical analysis of the material, giving the minimum percentages of CaO and SO_3, also the net weight of package. About the only raw gypsum shipped in packages is land plaster or agricultural gypsum; therefore, it should not be required to show on each tag the size and form of the material. Instead this could better be provided for under the heading of description of material. As to the chemical analysis of the material, this is so seldom required that it should not be required to list it. However, because some state laws may require additional information, it was thought advisable to call attention to this in a note. Practically all state laws require, in addition to the net weight, the gross weight of the package. Committee C-11 accordingly presents for publication as tentative the following revisions of Standard Specifications C 22:

Section 1 (*b*).—Change the minimum requirement for $CaSO_4 \cdot H_2O$ from "64.5 per cent" to read "75 per cent."

Section 7.—Change Paragraph (*b*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*b*) When shipped in packages for resale, the following information shall be legibly marked on each package or on a tag of suitable size attached thereto:
>
> Name of manufacturer,
> [Form and size of material,]
> *Description of material,*
> [Chemical analysis of the material, giving the minimum percentages of CaO and SO_3,]
> Net *and gross* weights of package.

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[2] 1939 Book of A.S.T.M. Standards, Part II, pp. 70, 73, 75, 67, 65.

[3] *Ibid.*, p. 77.

[4] *Ibid.*, p. 63.

Add a note following Paragraph (*c*) to read as follows:

NOTE.—State laws may require additional information.

III. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Keene's Cement (*C 61 – 30*).[5]—The committee recommends that the revision of Specifications C 61 published as tentative in 1938[6] be approved for reference to letter ballot of the Society for adoption as standard, with a modification in Section 8. This section requires the modified Vicat needle to give a penetration of 20 ± 2 mm. Tests conducted by several members of the committee indicated no appreciable difference in the strength of Keene's cement poured at consistencies of 20-mm. and 30-mm. penetration and inasmuch as the Federal Specification for Keene's Cement prescribes a 30-mm. penetration, it is recommended that the requirement in Section 8 for a penetration of "20 mm." be changed to read "30 mm."

Standard Methods of Testing Gypsum and Gypsum Products (*C 26 – 39*).[7]—The committee recommends that the revision of Methods C 26 published as tentative last year[8] be approved for reference to letter ballot of the Society for adoption as standard.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 22 members; 22 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE STANDARD			
Definitions of Terms Relating to Gypsum (C 11 - 31 T)....	21	0	1
II. REVISIONS OF STANDARDS			
Spec. for Gypsum Plasters (C 28 - 39; ASA A49. 3-1939), immediate adoption.........	20	0	2
Spec. for Gypsum Molding Plaster (C 59 - 30; ASA A49.4-1933), immediate adoption	18	0	4
Spec. for Gypsum Pottery Plaster (C 60 - 30; ASA A49.5-1933), immediate adoption....................	18	0	4
Spec. for Keene's Cement (C 61 - 30), immediate adoption....................	19	0	3
Spec. for Calcined Gypsum for Dental Plasters (C 72 - 30), immediate adoption....	18	0	4
Spec. for Gypsum Lath (C 37 - 34), immediate adoption....	21	0	1
Spec. for Gypsum (C 22 - 25; ASA A49.1-1933)............	21	0	1
III. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Spec. for Keene's Cement (C 61 - 30), as revised	19	0	3
Testing Gypsum and Gypsum Products (C 26 - 39).........	20	0	2

This report has been submitted to letter ballot of the committee which consists of 22 members; 22 members returned their ballots, of whom 21 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. S. WELLS,
Chairman.

H. J. SCHWEIM,
Secretary.

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[5] 1939 Book of A.S.T.M. Standards, Part II, p. 67.
[6] *Ibid.*, p. 1179.
[7] *Ibid.*, p. 86.
[8] *Ibid.*, p. 1179.

REPORT OF COMMITTEE C-12

ON

MORTARS FOR UNIT MASONRY

Committee C-12 on Mortars for Unit Masonry has held three meetings during the past year: in Atlantic City, N. J. on June 28, 1939, and in Washington, D. C., on December 11, 1939, and on March 22, 1940.

The membership at present totals 35, of whom 15 are classified as producers, 9 as consumers, and 11 as general interest members.

In addition to the standing subcommittees and working subcommittees listed in the 1939 report,[1] the following have been established:

Subcommittee V on Admixtures for Mortar (W. C. Voss, chairman),

Working Subcommittee on Mortar for Glass Block (G. M. Rapp, chairman), and

Joint Working Subcommittee (with Committee C-15) on Efflorescence (Frederick Heath, Jr., chairman).

Although no specifications and methods of test are being offered for publication as tentative at this time, the subcommittees have been quite active and two papers by a member of the committee will present in part data and tests which are being considered by the committee.

Subcommittee II on Methods of Test (F. O. Anderegg, chairman), acting through its Working Subcommittee on Workability and Plasticity (L. A. Wagner, chairman), made a study of the 10-in. flow table which included cooperative tests using a "calibrating mortar" prepared and distributed by P. S. Roller. A paper entitled "Some Characteristics of the Flow Table for Testing Mortar Consistency"[2] which makes recommendations concerning the construction and design of the flow table has been prepared by Mr. Anderegg. Another paper by Mr. Anderegg entitled "Some Properties of Mortar in Masonry" provides a detailed description of certain new techniques for testing mortar which he developed and which are being studied by the committee. The latter paper is to be presented at this annual meeting of the Society.[3]

The Working Subcommittee on Soundness and Volume Change of Mortar (R. E. Davis, chairman), under the joint jurisdiction of Subcommittees II and III, has prepared and presented a program for research on shrinkage and expansion of mortar. The committee is giving consideration to ways and means of carrying through this program.

The Working Subcommittee on Mortar for Glass Block (G. M. Rapp, chairman), also under the joint jurisdiction of Subcommittees II and III, has drafted complete specifications and methods of test for mortar for glass block which have been distributed in Committee C-12 for comment and criticism.

Subcommittee III on Specifications for Mortar (T. I. Coe, chairman) completed a rather extensive series of cooperative tests on masonry cement and cement-lime mortars. These tests indicated (1) the range in variation in such properties as compressive strength and water retentivity resulting from commercial variation in cements, limes, and sands, and (2) the variation in results caused by variation in methods of test from laboratory to laboratory. The "Primer on Specifications and Tests for Masonry Mortars" has been extensively revised by a special subcommittee (H. C. Plummer, chairman) and is being further considered by Committee C-12.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 371 (1939).

[2] To be published in the ASTM BULLETIN.

[3] See p. 1130.

Adoption as standard of the Tentative Specifications for Aggregate for Masonry Mortar (C 144 - 39 T) is not requested at this time as the committee feels that more experience with this type of specification is required.

Subcommittee V on Admixtures for Mortar (W. C. Voss, chairman), has considered a definition for admixtures.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 35 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. W. MCBURNEY,
Chairman.

H. C. PLUMMER,
Secretary.

REPORT OF COMMITTEE C-13

ON

CONCRETE PIPE

Committee C-13 on Concrete Pipe held a meeting at Chicago, Ill., on April 10, 1940, and at that time action was taken by the committee to recommend to the Society tentative revisions in the following standard specifications that are under the jurisdiction of the committee:

Standard Specifications for:

Concrete Sewer Pipe (C 14 – 35),

Reinforced Concrete Sewer Pipe (C 75 – 35), and

Reinforced Concrete Culvert Pipe (C 76 – 37).

Unfortunately, lack of time prevented the printing of these tentative revisions for formal consideration at the annual meeting of the Society and therefore they will be presented to the Society subsequent to the meeting through Committee E-10 on Standards.[1] This progress report presents a general summary of the revisions that are proposed.

Many of the changes are of an editorial nature designed to bring into agreement corresponding requirements in the three specifications which, in the interests of uniformity, should be identical in wording.

In the case of the Standard Specifications for Concrete Sewer Pipe (C 14 – 35) the proposed revision provides for an increase in the strength requirements to bring the specifications into agreement with the Federal Specification for Non-Pressure, Non-Reinforced Concrete Pipe (SS-P-371).

The revision of the Standard Specifications for Reinforced Concrete Sewer Pipe (C 75 – 35) will provide additional requirements for pipe 12, 15, 18, and 21 in. in internal diameter.

In the Standard Specifications for Reinforced Concrete Culvert Pipe (C 76 – 37) the revision includes changes in the requirements for 4500-lb. concrete in Table I, and the shell thickness of 18-in. pipe is to be changed from $2\frac{1}{4}$ in. to 2 in. These specifications are also being revised by the addition of pipe of larger diameter. In the present standard, the maximum size of pipe covered is 84 in. in internal diameter. In the revised specifications the maximum diameter will be 108 in. In the case of 30-in. pipe, the present requirements in Tables I and II for single-line reinforcement are to be changed to two lines of reinforcement.

The above are the more important revisions, other than those of an editorial nature, that are to be recommended. Due to the extensive nature of the changes the tentative revisions of these three standards will be issued as separate tentative specifications[1] to supersede when adopted as standard the present standard specifications.

The present officers of the committee were re-elected for the ensuing term of two years.

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[1] See Editorial Note, p. 275.

This report has been submitted to letter ballot of the committee which consists of 26 members; 24 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

T. F. DOLL,
Chairman.

M. W. LOVING,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee C-13 presented to the Society through Committee E-10 on Standards tentative revisions of the Standard Specifications for Concrete Sewer Pipe (C 14 – 35), for Reinforced-Concrete Sewer Pipe (C 75 – 35), and for Reinforced-Concrete Culvert Pipe (C 76 – 37). These revisions, in the form of new tentative specifications, were accepted by Committee E-10 on August 28, 1940, and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 235, 243, and 253.

REPORT OF COMMITTEE C-15

ON

MANUFACTURED MASONRY UNITS

Committee C-15 on Manufactured Masonry Units held three meetings during the year: in Atlantic City, N. J., on June 28, 1939, during the annual meeting of the Society, and in Washington, D. C., on December 12, 1939, and on March 21, 1940. Regular business was transacted at two of the meetings; the third meeting was devoted to a discussion of research on masonry units at which the following papers were presented and discussed:

The Properties of Concrete Masonry and Masonry Units, by P. M. Woodworth,

The Frost Resistance of Clay Building Brick, by J. W. McBurney,

Specifications for Clay Building Brick, by J. H. Hansen,

Research on the Factors Affecting the Durability of Clay Brick, by H. G. Schurecht, and

Efflorescence on Masonry, by Frederick Heath, Jr.

Subsequent to the 1939 annual meeting Committee C-15 presented to the Society through Committee E-10 on Standards the following recommendations which were accepted[1] by Committee E-10 on August 24, 1939, and appear in the 1939 Book of A.S.T.M. Standards:[2]

Revision of Tentative Specifications for Sewer Brick (Made from Clay or Shale) (C 32 - 39 T),

Revision of Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 - 39 T), and

Tentative Revision of Standard Methods of Sampling and Testing Brick (C 67 - 39) in the form of new Tentative Method of Freezing-and-Thawing Test of Brick (C 67 - 39 T), to be added when adopted to the present standard.

With deep regret the committee records the loss through death of a long-time member, George F. Schlesinger, Chief Engineer and Managing Director of the National Paving Brick Assn. Mr. Schlesinger had been very helpful in the activities of the committee and his loss is keenly felt.

As the result of resignations, deaths, and the election of new members the membership of the committee now totals 56, of whom 24 are producers, 13 consumers, and 19 general interest members.

I. REVISION OF TENTATIVE STANDARD

In order to avoid the possibility of misinterpretation, but without changing the intent of the specifications, the committee recommends that the Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 - 39 T)[3] be revised as follows and continued as tentative:

Section 2 (d).—At the end of the first paragraph, which presents the tabulation of the freezing-and-thawing test requirements, add a footnote to read as follows:

> Brick are not required to conform to the provisions of Section 2 (*d*), and these do not apply unless the sample fails to conform to the requirements for absorption and saturation coefficient prescribed in Table I as well as the strength and absorption requirements in Section 2 (*c*).

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee C-15 reported the following results of the letter ballot vote of a total of 40 ballots returned from a committee membership of 57: C 32 - 37 T, affirmative 30, negative 3, ballots marked "not voting" 7; C 62 - 37 T, affirmative 33, negative 1, ballots marked "not voting" 6; C 67 - 39, affirmative 36, negative 0, ballots marked "not voting" 4.

[2] 1939 Book of A.S.T.M. Standards, Part II, pp. 925, 916, 930.

[3] *Ibid.*, p. 916.

II. ADOPTION OF TENTATIVE STANDARD AS STANDARD

As no criticisms of the Tentative Specifications for Solid Load-Bearing Concrete Masonry Units (C 145 - 39 T)[4] have been received, it is recommended that the specifications be approved for reference to letter ballot of the Society for adoption as standard.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 56 members; 42 members returned their ballots, the results being shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE STANDARD			
Spec. for Building Brick (Made from Clay or Shale) (C 62 - 39 T)	37	1	4
II. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Spec. for Solid Load-Bearing Concrete Masonry Units (C 145 - 39 T)	40	0	2

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Clay Building Brick (J. W. McBurney, chairman).—Investigations of the factors affecting the resistance of clay building brick to frost action have been continued in the laboratories of the National Bureau of Standards and Alfred University. Effects of temperatures during freezing on the rate of disintegration of bricks were studied and data were obtained on the relations between the resistance of brick to frost action and physical properties which are determined easily in the laboratory.

Subcommittee III on Concrete Units (W. G. Kaiser, chairman).—The research project on the properties of concrete masonry units, which has been under way at the University of Wisconsin, was completed and the results are now available in a paper entitled "Tests on Concrete Masonry Units Using Tamping and Vibration Molding Methods."[5] No revisions of existing standards and no new tentative standards are being presented this year, but the subcommittee is following with interest new developments in the field which are expected to result in a demand for standards for new products.

Subcommittee V on Paving Brick (F. H. Jackson, chairman).—The investigation on paving brick being conducted jointly by the Ohio State Highway Commission and the Public Roads Administration is expected to furnish information which will serve as a basis for revising the Tentative Specifications for Paving Brick (C 7 - 38 T). Three miles of paving constructed of different kinds of brick are under observation. Elaborate records are being taken to determine the service performance of the brick and the relation between performance and properties which can be determined easily and quickly. The committee is recommending that Specifications C 7 - 38 T be continued as tentative until the data from the foregoing investigation become available. A minor revision to make the specifications conform with general practice and the Simplified Practice Recommendation R 1 - 40 of the U. S. Department of Commerce is to be submitted to the Society subsequent to the annual meeting.[6]

Subcommittee VII on Glazed Brick and Tile (J. R. Kauffman, chairman).—Although there appears to be a need for revising the Tentative Specifications for Glazed Building Units (C 126 - 39 T), the industry has been slow in sponsoring investigations for the purpose of obtaining the needed information. It is hoped that the glazed brick and tile industry and the architectural terra cotta industry will sponsor such an investigation in the coming year.

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[4] 1939 Book of A.S.T.M. Standards, Part II, p. 933.

[5] *Proceedings*, Am. Concrete Inst., Vol. 36, p. 121 (1940).

[6] See Editorial Note, p. 278.

Subcommittee VIII on Structural Clay Tile (H. C. Plummer, chairman).—In response to a newly developed demand, the subcommittee is undertaking studies which eventually are expected to lead to the preparation of specifications for smooth facing tile. These are unglazed units designed for use in interior and exterior unplastered walls of buildings and are usually of a finer texture than many of the commercial structural tile.

Joint Working Subcommittee (with Committee C-12) on Efflorescence (Frederick Heath, Jr., chairman).—This subcommittee has reviewed much of the literature relating to efflorescence on masonry, paying especial attention to methods of determining whether masonry materials are likely to be a source of efflorescence.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 56 members; 44 members returned their ballots, of whom 43 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of of the committee,

D. E. PARSONS,
Chairman.

J. W. WHITTEMORE,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee C-15 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Specifications for:
  Paving Brick (C 7 – 38 T),
Tentative Revisions of Standard:
  Specifications for Structural Clay Load-Bearing Wall Tile (C 34 – 39),
  Specifications for Structural Clay Non-Load-Bearing Tile (C 56 – 39), and
  Methods of Sampling and Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (C 67 – 39).

These recommendations were accepted by Committee E-10 on August 28, 1940, and the revised tentative specifications and tentative revisions appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 183, and 338.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 27, 1939, in Atlantic City, N. J., during the annual meeting of the Society, and on April 12, 1940, in Cincinnati, Ohio, during the meeting of the American Chemical Society.

The committee recommends that the Society continue as a member-body in the Inter-Society Color Council, with M. Rea Paul, an official delegate of the Society, representing the committee.

Committee D-1 has, during the past year, arranged for the presentation of two papers and one motion picture at its meetings, as follows:

"The Status of Linseed Oil,"[1] by R. D. Bonney,

"Liquid Driers—Their Function, Difficulties Involved, Improvements, and Methods of Test,"[2] by W. T. Pearce, and

A motion picture containing scenes taken at the research and development laboratories and manufacturing plants of the American Cyanamid and Chemical Corp.

During the past year a new subcommittee was appointed, designated Subcommittee II on Drying Oils, under the chairmanship of Francis Scofield. Subcommittee III, which had previously carried on this work under the title of Testing of Paint Vehicles, was discontinued. Subcommittee XXVI on Underground Pipe Protection was also discontinued, and its chairman, F. N. Speller, was appointed adviser on all matters in this connection.

During the year, on the recommendation of Committee D-1, seven standards were submitted by the Society to the American Standards Association and approved as American standards.

Since the last annual meeting, 20 new members have been elected, 9 resignations have been accepted, leaving a total membership of 235.

Of the one adviser and fourteen D-1 subcommittees, four furnished reports of progress and eight submitted detailed reports affecting standards, which have resulted in the committee making the recommendations given below to be acted upon at the annual meeting.

In this report, recommendations affecting standards and tentative standards are noted in brief form below, together with the results of the letter ballot. These recommendations are explained in the reports of the subcommittees directly responsible for them.

I. New Tentative Standards

The committee submits for publication as tentative the following three new specifications, three methods of test, and definitions of terms:

Tentative Specifications for:

Titanated Lithopone, to be added, when adopted as standard, to the Standard Specifications for Zinc Sulfide Pigments (D 477 – 39), proposed by Subcommittee XV,

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[1] ASTM Bulletin, No. 100, October, 1939, p. 27.

[2] *Ibid.*, p. 31.

Ultramarine Blue, to replace immediately the Standard Specifications for Ultramarine Blue (D 262 – 28) which are recommended for withdrawal, proposed by Subcommittee XV, and

Carbon Black, proposed by Subcommittee XV.

Tentative Methods of:

Test for Consistency of Enamel Type Paints, proposed by Subcommittee VIII,

Test for Phthalic Anhydride Content of Alkyd Resin Solutions, proposed by Subcommittee IX, and

Testing Liquid Driers, proposed by Subcommittee IX.

Tentative Definitions of:

Terms Relating to Paint, Varnish, Lacquer, and Related Products to replace immediately the Standard Definitions of Terms Relating to Paint, (D 16 – 24) which are recommended for withdrawal, proposed by Subcommittee VI.

II. REVISIONS OF STANDARDS

The committee recommends for immediate adoption revisions in the following standards, and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for (proposed by Subcommittee XV):

Leaded Zinc Oxide (D 80 – 24),
Mineral Iron Oxide (D 84 – 27),
Ocher (D 85 – 27),
Chrome Yellow (D 211 – 27),
Pure Chrome Green (D 212 – 27),
Reduced Chrome Green (D 213 – 27),
Prussian Blue (D 261 – 39),
Chrome Oxide Green (D 263 – 28),
Reduced Para Red (D 264 – 39),
C. P. Para Red Toner (D 475 – 39), and
C. P. Zinc Yellow (Zinc Chromate) (D 478 – 39).

Standard Methods of:

Sampling and Analysis of Shellac (D 29 – 39), proposed by Subcommittee XIII, and

Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 37), proposed by Subcommittee XXV.

The committee further recommends that the revisions proposed in the following standards be accepted for publication as tentative:

Standard Methods of:

Chemical Analysis of White Linseed Oil Paints (D 215 – 37), proposed by Subcommittee VIII, and

Sampling and Testing Turpentine (D 233 – 36), proposed by Subcommittee XII.

III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard, and that the one so indicated be revised as referred to in the report of the subcommittee:

Tentative Specifications for:

Zinc Dust (Metallic Zinc Powder) (D 520 – 39 T), proposed by Subcommittee XV.

Tentative Methods of:

Chemical Analysis of Zinc Dust (Metallic Zinc Powder) (D 521 – 39 T), revised as proposed by Subcommittee VIII, and

Test for Reactivity of Paint Liquids (D 479 – 38 T), proposed by Subcommittee IX.

IV. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

The committee recommends that the tentative revisions of the following five standards be approved without change for reference to letter ballot of the Society for adoption as standard, as proposed by Subcommittee XXV:

Standard Specifications for:

Normal Butyl Acetate (88 to 92 per cent Grade) (D 303 – 33),

Butanol (Normal Butyl Alcohol) (D 304 – 33),

Amyl Alcohol (Synthetic) (D 319 – 33),

Butyl Propionate (90 to 93 per cent Grade) (D 320 – 33), and

Ethyl Lactate (Synthetic) (D 321 – 33).

V. WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the Standard Specifications for

Ultramarine Blue (D 262 – 28), proposed by Subcommittee XV, and Standard Definitions of Terms Relating to Paint (D 16 – 24), proposed by Subcommittee VI, for the reasons referred to in the reports of the subcommittees.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Titanated Lithopone, to be added, when adopted, to Standard Spec. D 477	67	1	36
Spec. for Ultramarine Blue, to replace Standard Spec. D 262	61	0	43
Spec. for Carbon Black	57	0	47
Test for Consistency of Enamel Type Paints	74	0	30
Test for Phthalic Anhydride Content of Alkyd Resin Solutions	56	2	46
Testing Liquid Driers	67	4	33
Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products, to replace Standard Definitions D 16 – 24	114	1	9
II. REVISIONS OF STANDARDS			
Spec. for Leaded Zinc Oxide (D 80 – 24), immediate adoption	66	1	37
Spec. for Mineral Iron Oxide (D 84 – 27; ASA K25—1937) immediate adoption	59	0	45
Spec. for Ocher (D 85 – 27), immediate adoption	58	0	46
Spec. for Chrome Yellow (D 211 – 27; ASA K27– 1937) immediate adoption.	70	0	34
Spec. for Pure Chrome Green (D 212 – 27), immediate adoption	69	0	35
Spec. for Reduced Chrome Green (D 213 – 27; ASA K28—1937) immediate adoption	68	0	36
Spec. for Prussian Blue (D 261 – 39; ASA K29—1939) immediate adoption	70	0	34
Spec. for Chrome Oxide Green (D 263 - 28; K37– 1937), immediate adoption	60	0	44
Spec. for Reduced Para Red (D 264 – 39; ASA K31—1939) immediate adoption	66	0	38
Spec. for C.P. Para Red Toner (D 475 – 39), immediate adoption	68	0	36
Spec. for C.P. Zinc Yellow (Zinc Chromate) (D 478 – 39), immediate adoption	65	0	39
Sampling and Analysis of Shellac (D 29 – 39), immediate adoption	46	0	58
Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 37), immediate adoption	53	0	51
Chemical Analysis of White Linseed Oil Paints (D 215 – 37)	73	1	30
Sampling and Testing Turpentine (D 233 - 36; ASA K33—1937)	52	3	49
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Zinc Dust (Metallic Zinc Powder) (D 520 – 39 T)	54	0	50
Chemical Analysis of Zinc Dust (Metallic Zinc Powder) (D 521 – 39 T), as revised	57	0	47
Test for Reactivity of Paint Liquids (D 479 – 38 T)	70	0	34
IV. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Spec. for Normal Butyl Acetate (88 to 92 per cent Grade) (D 303 – 33)	45	0	59
Spec. for Butanol (Normal Butyl Alcohol) (D 304 – 33)	46	0	58
Spec. for Amyl Alcohol (Synthetic) (D 319 – 33)	46	0	58
Spec. for Butyl Propionate (90 to 93 per cent Grade) (D 320 – 33)	45	0	59
Spec. for Ethyl Lactate (Synthetic) (D 321 – 33)	44	0	60
V. WITHDRAWAL OF STANDARD			
Spec. for Ultramarine Blue (D 262 - 28; ASA K30—1937)	59	0	45
Definitions of Terms Relating to Paint (D 16 – 24)	111	0	13

VI. EDITORIAL CHANGE IN STANDARD

An editorial change is being made in the Standard Specifications for Petroleum Spirits (Mineral Spirits) (D 235 – 39) as proposed by Subcommittee XI.

These recommendations have been submitted to letter ballot of the committee which consists of 235 members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Underground Pipe Protection (F. N. Speller, adviser).—The American Petroleum Institute's underground coating tests are now being inspected for the last time, after ten years' exposure, and the results will be made available in the near future.

Subcommittee II recommends that the Tentative Specifications for Raw Soybean Oil (D 124 – 37 T) be included in the letter ballot in the spring of 1941 for adoption as standard. This recommendation was accepted by vote of Committee D-1.

Subcommittee VI on Definitions (C. H. Rose, chairman).—This subcommittee recommends for withdrawal the Standard Definitions of Terms Relating to Paint (D 16 – 24), since revisions in some definitions are being considered and new definitions are in preparation. This subcommittee further recommends for publication as tentative, Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products.[3]

Subcommittee VI has under consideration the preparation of suitable definitions for the following terms: surfacer, putty, glaze, japan, gloss oil, flatting agent, lacquer, resin, natural resin, fossil resin, synthetic resin, alkyd resin, baking, baking temperature, forced drying temperature, and baked finish.

Subcommittee VII on Accelerated Tests for Protective Coatings (H. A. Nelson, chairman).—This subcommittee reports progress in the work of its subgroups.

Group 1 on House Paints (C. H. Rose, chairman) is continuing its investigations of accelerated testing of house paints for color retention. Additional cooperative tests are being planned, based on the 1938 test experiences. Thus far, the most satisfactory of the test cycles includes rest periods and a periodic conditioning of the panels at 32 F. Standards by which to judge the accelerated tests will be established by exposing the same paints at various locations.

Group 2 on Oleoresinous Type Enamels (C. Y. Hopkins, chairman) has completed a series of accelerated weathering and outdoor tests on enamels, the results of which are presented in a report appended hereto.[4]

Group 6 on Metal Protective Paints (J. C. Moore, chairman) is endeavoring to predict, by accelerated testing means, results that Subcommittee XXIX will secure in its study of pretreatments on panels exposed at Sayville, L. I., and Bethlehem, Pa. No definite conclusions have been drawn. Tests of other than accelerated weathering types are also being conducted. Work is now in progress on the development of reference standards illustrating various degrees of rusting of painted steel or iron surfaces.

Subcommittee VII has decided to prepare procedures for certain phases of accelerated testing, to include test panels for different classes of materials to be followed by methods of an accelerated nature that are deserving of consideration.

H. G. Arlt has been appointed chairman of a group to prepare a questionnaire that will be employed to secure information regarding test methods now in use in the paint industry for evaluating the service characteristics of organic finishes.

Subcommittee VIII on Methods of Analysis of Paint Materials (E. F. Hickson, chairman).—This subcommittee recommends for publication as tentative a new Method of Test for Consistency of Enamel Type Paints.[5] It is pointed out that the Society has no suitable method for measuring the consistency of paints, and since there have been many requests for such a method, the one proposed has been developed to meet this need.

The subcommittee further recommends the publication of the report of its Work Committee, appended hereto, together with the method of test for consistency of enamel type paints originally proposed by that committee.

The subcommittee recommends the adoption as standard of the Tentative Methods of Chemical Analysis of Zinc

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[3] These definitions were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 318.

[4] See p. 289.

[5] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 316.

Dust (Metallic Zinc Powder) (D 521 - 39 T),[6] with the following revision:

Section 5.—In Paragraph (*a*) include the following description of three additional solutions, and change the procedure in Paragraph (*b*) to read as follows:

> *Sodium Acetate Solution* (*20 per cent*).
>
> *Ferric Chloride Solution.*—Prepare a solution containing 20 g. of $FeCl_3 \cdot 6H_2O$ and 20 ml. of the $NaC_2H_3O_2$ solution per 100 ml. It is advisable to make up only one day's supply at a time (50 ml. for each determination).
>
> *Zimmerman-Reinhardt Solution.*—Prepare a solution containing 67 g. of $MnSO_4 \cdot 4H_2O$, 130 ml. of H_2SO_4 (sp. gr. 1.84), and 138 ml. of H_3PO_4 (sp. gr. 1.7) per liter.
>
> (*b*) *Procedure.*[a]—Weigh accurately approximately 0.2 g. of the sample, transfer immediately to a dry, 600-ml. Erlenmeyer heavy-wall flask, and add 50 ml. of the $FeCl_3$ solution. Tightly stopper the flask, and agitate constantly for approximately 15 min. As soon as the zinc dust is all dissolved, add 50 ml. of the Zimmerman-Reinhardt solution and 250 ml. of water, and titrate with the standard $KMnO_4$ solution. Correct for a blank titration (usually 0.1 to 0.2 ml.). One milliliter of 0.1 *N* $KMnO_4$ is equivalent to 0.00327 g. of zinc. Calculate metallic zinc according to the following formula:
>
> Metallic zinc, per cent =
>
> $$\frac{\text{ml. of } KMnO_4 \times 0.00327}{\text{wt. of sample}} \times 100$$
>
> NOTE.—If preferred, the reduced iron may be titrated with a 0.1 *N* standard solution of $K_2Cr_2O_7$ using sodium diphenylamine sulfonate in the presence of H_3PO_4 as an internal indicator.[b]
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>
> [a] This is a rapid method intended for routine analysis. The results are inclined to be somewhat low, and for highest accuracy and particularly for referee tests, the hydrogen evolution method should be used. See *Proceedings*, Am. Soc. Testing Mats., Vol. 18, Part II, p. 220 (1918).
>
> [b] See "Textbook of Quantitative Inorganic Analysis" by Kolthoff and Sandell, 1936, p. 579.

The subcommittee further recommends for publication as tentative a revision of the Standard Methods of Chemical Analysis of White Linseed Oil Paints (D 215 - 37),[7] as follows:

Section 31 (*b*).—In place of the first five sentences which read as follows:

> In the absence of ZnS or TiO_2 report $BaSO_4$ as $BaSO_4$. If ZnS is present, calculate the $BaSO_4$ equivalent by multiplying by 2.85; report sum of ZnS + $BaSO_4$ as "lithopone." If TiO_2 is present, calculate the $BaSO_4$ equivalent by multiplying by 3.17; report sum of TiO_2 + $BaSO_4$ as "titanium pigment." Report residual $BaSO_4$ as $BaSO_4$. If TiO_2 is present and $BaSO_4$ is absent or is present in a smaller amount than would be indicated by the above factor, then report TiO_2 as TiO_2, and $BaSO_4$ as $BaSO_4$.

substitute the following sentence:

> Report TiO_2 as TiO_2, ZnS as ZnS, and $BaSO_4$ as $BaSO_4$.

It is further recommended that the Tentative Methods of Sampling and Testing Aluminum Powder and Paste (D 480 - 38 T) remain in a tentative status, pending the completion of certain studies of tests for leafing characteristics.

Subcommittee IX on Varnish (W. T. Pearce, chairman).—This subcommittee recommends for publication as tentative a Method of Test for Phthalic Anhydride Content of Alkyd Resin Solutions.[8] Such solutions are being sold in increasing quantities to manufacturers of paints, enamels, and similar finishes. The Navy Department and many states have specifications covering this class of material. Methods of test for all of these require that the nonvolatile part contain a definite amount of phthalic anhydride, usually expressed as glyceryl phthalate. Methods of analysis and factors involved in making the calculations have been the subject of many discussions. In the case of several important transactions, rejections of shipments have been made because of misunderstandings as to methods of analysis. The proposed method of analysis and accurate method of calculation are considered satisfactory for use in the examination of shipments of this class of resin solutions.

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[6] 1939 Book of A.S.T.M. Standards, Part II, p. 1134.

[7] 1939 Book of A.S.T.M. Standards, Part II, p. 818.

[8] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 315.

Subcommittee IX also recommends for publication as tentative new Methods of Testing Liquid Driers.[9] It is pointed out that large quantities of liquid driers are purchased by the Federal Government, prominent railroads, paint manufacturers, and most painters. During recent years, considerable improvement has been made in such driers. In spite of the large quantities of such materials purchased, the Society has no specifications relating to them. The purpose for which a finish is used determines to a degree the type of drier desired. At the present time, consumers of these materials do not have available specifications which would enable them to secure the type of drier needed. To develop such specifications for the several types of liquid driers now being used, accurate methods of test and analysis were required. Samples of typical driers have been studied by several methods, and those proposed in the new methods of test were found to be satisfactory.

The development of specifications covering the principal types of driers that are purchased today is now under consideration.

This subcommittee recommends the adoption as standard of the Tentative Method of Test for Reactivity of Paint Liquids (D 479 - 38 T).[10]

A study is being carried on by this subcommittee of a method for evaluating the color of varnishes.

Subcommittee XI on Paint Thinners Other Than Turpentine (M. B. Chittick, chairman).—On the recommendation of this subcommittee an editorial change is being made in the Standard Specifications for Petroleum Spirits (Mineral Spirits) (D 235 - 39).[11] In the table of requirements in Section 2 the term "dry point" is being changed to read "end point."

Subcommittee XII on Turpentine (C. F. Speh, chairman).—This subcommittee recommends for publication as tentative, revisions in the Standard Methods of Sampling and Testing Turpentine (D 233 - 36),[12] as follows:

Color Test.—Add as a new Section 5 the following description of the colorimeter tube, renumbering the subsequent sections accordingly:

> 5. *Turpentine Colorimeter Tube.*—The colorimeter tube shall be a straight, cylindrical tube of clear, colorless, soda-lime glass. It shall be 230 to 280 mm. in length, 21 ± 1 mm. in inside diameter, and $1\frac{1}{4}$ to $1\frac{1}{2}$ mm. in wall thickness. The bottom may be (*1*) blown with the tube, of a uniform thickness of 1.0 to 1.5 mm., with the outer surface flat-ground and polished, and showing little or no distortion of the field of view when the tube is filled to a depth of 50 mm. with turpentine; or it may be (*2*) a separate colorless glass plate vitreously fused or sealed to the bottom of the cylinder, 1.0 to 1.5 mm. in thickness, with the outer surface flat-ground and polished, and having a diameter not more than 1 mm. greater than the outside diameter of the cylinder. Graduation lines shall be ruled or etched on the outside of the cylinder at each 2 mm. from the level of the inner surface of the bottom, each fifth line being longer, and each tenth (20-mm.) line still longer and marked to show the distance from the bottom.

Section 9 (*d*).—Specifications for a new turpentine distillation thermometer have been prepared and it is accordingly proposed to change this paragraph from its present form to read as follows:

> (*d*) *Thermometer.*—An A.S.T.M. turpentine distillation thermometer, 3 in. partial immersion, having a range of 148 to 182 C.,

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[9] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 311.

[10] 1939 Book of A.S.T.M. Standards, Part II, p. 1139.

[11] 1939 Book of A.S.T.M. Standards, Part II, p. 716.

[12] *Ibid.*, p. 726.

conforming to the requirements for this thermometer as prescribed in the Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1 - 40 T),[a] of the American Society for Testing Materials[13] shall be used.

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[a] The short total immersion turpentine distillation thermometer, having a range of 145 to 200 C., conforming to the requirements for this thermometer as previously prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1 - 39) of the American Society for Testing Materials,[14] may be used in lieu of the new standard 3-in. partial immersion turpentine distillation thermometer, if desired.

Subcommittee XIII on Shellac (W. H. Gardner, chairman).—This subcommittee recommends for immediate adoption a revision in the Standard Methods of Sampling and Analysis of Shellac (D 29 - 39),[15] as follows:

Section 31.—Change from its present form to read as follows:

> 31. Weigh a quantity of the shellac varnish that will contain approximately 10 g. of nonvolatile matter, and place in a 200 ml. tall form beaker to which add 2.5 g. of sodium carbonate and 150 ml. of hot water. Immerse the beaker in a steam or boiling-water bath and stir until the shellac is in solution. Then cover with a watch glass and complete the determination in accordance with the procedure described in Section 20.

Subcommittee XIII has successfully developed a suitable method for the determination of the color of orange shellac, and recommends that it be published in the ASTM BULLETIN for the purpose of soliciting further suggestions and comments prior to its presentation to the Society for publication as tentative.

A review has been concluded by this subcommittee of the draft British Standard Specification for Lac (CF (B) 2368), and a statement has been prepared containing recommendations and criticisms that has been approved by Committee D-1 for transmittal by the Society to the British Standards Institution.

The subcommittee recommends that the Tentative Specifications for Shellac Varnishes (D 360 - 39 T) remain in a tentative status for the coming year.

This subcommittee is considering the development of specifications for a dewaxed orange shellac dissolved in isopropyl alcohol.

Subcommittee XV on Specifications for Pigments Dry and in Oil when Marketed in That Form (C. L. Crockett, chairman).—This subcommittee recommends for publication as tentative, Specifications for Titanated Lithopone,[16] which are to be added, when adopted, to the Standard Specifications for Zinc Sulfide Pigments (D 477 - 39). It is pointed out that titanated lithopone has now become sufficiently well established and is sold in such quantities as to justify specifications covering its composition and properties.

Subcommittee XV also recommends for publication as tentative revised Specifications for Ultramarine Blue,[17] to replace immediately the Standard Specifications for Ultramarine Blue (D 262 - 28) which do not adequately meet present-day requirements and are accordingly recommended for withdrawal.

New Specifications for Carbon Black[18] are also recommended for publication as tentative. There are now A.S.T.M. standard specifications covering both both bone black (D 210 - 30) and lampblack (D 209 - 30) for paint pigment purposes. As there are no A.S.T.M. specifications for carbon black, which is thought to be equally as important for paint pigment purposes, a new tentative standard is being proposed. It is also believed that these new specifications would define the grade of carbon black that is used for paint pigment purposes, as differentiated from those grades employed in rubber and other commodities.

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[13] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 321.

[14] 1939 Book of A.S.T.M. Standards, Part II, p. 838

[15] 1939 Book of A.S.T.M. Standards, Part II, p. 748.

[16] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 310.

[17] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 308.

[18] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 307.

A revision is proposed for immediate adoption in the Standard Specifications for Leaded Zinc Oxide (D 80 - 24). It is pointed out that for some time many combinations of zinc oxide and basic lead sulfate have been produced, some of which contained as much as 75 per cent or more of normal or basic lead sulfate, whereas the existing standard covers only two combinations, known as high-leaded and low-leaded, of which the former grade limits the lead content to less than 40 per cent. Under these conditions the present specifications are not sufficiently broad to cover materials being offered to the trade, and are therefore being revised.[19]

Adoption as standard, without revision, is recommended in the case of the Tentative Specifications for Zinc Dust (Metallic Zinc Powder) (D 520 - 39 T).[20]

Subcommittee XV further proposes for immediate adoption revisions in a number of the standard specifications for colored pigments. The revisions involve the addition of requirements for moisture and other volatile matter present in the pigments both in the dry form and as a paste in oil. The earlier specifications usually included a requirement for moisture in the pigment in paste form, but not in the dry pigment, which is its principal source. Moisture requirements appearing in these specifications are not consistent with those actually found in the products offered to the trade. In view of these considerations, the subcommittee recommends that the following standard specifications be revised as indicated in detail below:

Standard Specifications for Mineral Iron Oxide (D 84 - 27):[21]

Section 2.—In Paragraph (*a*) add the following requirement: "Moisture and other volatile matter, 2.0 per cent, max."

In Paragraph (*b*) change the requirement for moisture and other volatile matter from "0.7 per cent, max.," to read "1.5 per cent, max."

Standard Specifications for Ocher (D 85 - 27):[21]

Section 2.—In Paragraph (*a*) add the following requirement: "Moisture and other volatile matter, 1.0 per cent, max."

In Paragraph (*b*) change the requirement for moisture and other volatile matter from "0.5 per cent, max." to read "0.7 per cent, max."

Standard Specifications for Chrome Yellow (D 211 - 27):[22]

Section 2.—In Paragraph (*a*) add the following requirement: "Moisture and other volatile matter, 1.0 per cent, max."

In Paragraph (*b*) change the requirement for moisture and other volatile matter from "0.7 per cent, max." to read "1.0 per cent, max."

Standard Specifications for Pure Chrome Green (D 212 - 27):[23]

Section 2.—In Paragraph (*a*) add the following requirement: "Moisture and other volatile matter, 4.0 per cent, max."

In Paragraph (*b*) add the following requirement: "Moisture and other volatile matter, 3.0 per cent, max."

Standard Specifications for Reduced Chrome Green (D 213 - 27):[24]

Section 2.—In Paragraphs (*a*) and (*b*) add the following requirement: "Moisture and other volatile matter, 1.0 per cent, max."

Standard Specifications for Prussian Blue (D 261 - 39):[25]

Section 2.—In Paragraph (*b*) change the requirement for moisture and other volatile matter from "3.0 per cent, max." to read "3.5 per cent, max."

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[19] These specifications were adopted as standard by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 130.

[20] 1939 Book of A.S.T.M. Standards, Part II, p. 1129.

[21] 1939 Book of A.S.T.M. Standards, Part II, pp. 614, 616.

[22] *Ibid.*, p. 612.

[23] *Ibid.*, p. 607.

[24] *Ibid.*, p. 609.

[25] *Ibid.*, p. 621.

Standard Specifications for Reduced Para Red (D 264 – 39),[26] and for C.P. Para Red Toner (D 475 – 39):[27]

Section 2.—In Paragraph (*a*) add the following requirement: "Moisture and other volatile matter, 1.0 per cent, max."

Standard Specifications for C.P. Zinc Yellow (Zinc Chromate) (D 478 – 39):[28]

Section 2.—In Paragraph (*a*) add the following requirement: "Moisture and other volatile matter, 1.0 per cent, max."

In Paragraph (*b*) change the requirement for moisture and other volatile matter from "0.7 per cent, max." to read "0.8 per cent, max."

This subcommittee further recommends for immediate adoption a revision in the Standard Specifications for Chrome Oxide Green (D 263 – 28),[29] as given in detail below. It is pointed out that the chrome oxide green (dry pigment) now available to the trade has such high oil absorption that the present proportions of pigment and oil make a paste entirely too stiff for satisfactory use. In addition, a revision is proposed in the moisture content similar to that recommended above in the case of the other colored pigments.

Section 2.—In Paragraphs (*a*) and (*b*) add the following requirement: "Moisture and other volatile matter, 0.5 per cent, max."

In Paragraph (*b*) change the requirement for pigment from "85 per cent, min." to read "75 per cent, min.," and change the requirement for linseed oil from "15 per cent, max." to read "25 per cent, max."

Subcommittee XVIII on Physical Properties of Materials (M. Rea Paul, chairman).—This subcommittee recommends that the Tentative Method of Test for Specular Gloss of Paint Finishes (D 523 – 39 T) be continued in a tentative status for the coming year.

Progress is reported in the matter of studying a method for specifying a color that would be so readily understood that a person could easily visualize the appearance of the color from its notation.

Subcommittee XXV on Cellulose Ester Coatings (R. M. Carter, chairman).—This subcommittee recommends for immediate adoption a revision in the Standard Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 37),[30] as follows:

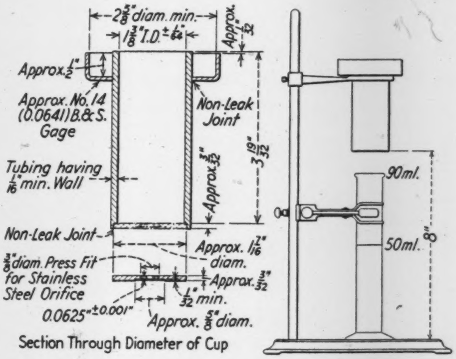

FIG. 1.—Consistency Cups and Apparatus Assembly for Testing Lacquers and Lacquer Enamels.

NOTE 1.—Four consistency cups constitute a set, as follows:

	Diameter of Orifice, in.
First cup	0.07 ± 0.0001
Second cup	0.10 ± 0.0001
Third cup	0.15 ± 0.0001
Fourth cup	0.25 ± 0.0001

NOTE 2.—Cups made of brass or bronze. Orifice disks made of 18 per cent chromium, 8 per cent nickel stainless steel. The diameter of the orifice pressed in the cup shall be stamped on the cup.

NOTE 3.—Fractional dimensions subject to permissible variations of plus or minus 0.01 in., unless otherwise specified.

Fig. 1.—In place of the present figure which illustrates a suitable design of the consistency cups and test assembly, substitute the revised illustration shown in the accompanying Fig. 1.

This subcommittee recommends that the existing tentative revisions[31] of the following standard specifications be approved for adoption as standard:

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[26] 1939 Book of A.S.T.M. Standards, Part II, p. 618.
[27] *Ibid.*, p. 620.
[28] *Ibid.*, p. 635.
[29] *Ibid.*, p. 611.

[30] 1939 Book of A.S.T.M. Standards, Part II, p. 801.
[31] *Ibid.*, pp. 1183, 1184.

Standard Specifications for:

- Normal Butyl Acetate (88 to 92 per cent Grade) (D 303 - 33),
- Butanol (Normal Butyl Alcohol) (D 304 - 33),
- Amyl Alcohol (Synthetic) (D 319 - 33),
- Butyl Propionate (90 to 93 per cent Grade) (D 320 - 33), and
- Ethyl Lactate (Synthetic) (D 321 - 33).

Subcommittee XXIX on Painting of Structural Iron and Steel (A. J. Eickhoff, chairman).—This subcommittee reports progress in its study to determine the amount of protection (freedom from rust) afforded by the presence or absence of mechanical or chemical treatment of steel surfaces prior to painting. Within the past few years there have appeared on the market a number of aqueous inhibitive solutions to be applied at atmospheric temperature. These solutions consist essentially of a mixture of water, chromates, reduced chromates, orthophosphoric acid, wetting agents, and metallic phosphates. This has given rise to the discussion concerning the relative advantages of painting over cold-pretreated surfaces, mill scale, sand-blasted steel, and surfaces which have rusted, been wire-brushed, and given a cold pretreatment. It is generally recognized that hot pretreatments now used in the automotive industry are not practical, from the standpoint of field application, for structural steel. An extensive series of tests on metal plates of these pretreatments that were subsequently painted are now being carried on.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 235 members; 104 members returned their ballots, of whom 77 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. E. SMITH,
Chairman.

M. REA PAUL,
Secretary.

ACCELERATED WEATHERING TESTS OF ENAMELS ON STEEL AND CORRELATION WITH OUTDOOR EXPOSURE

Report of Group 2 on Enamels of Subcommittee VII on Accelerated Tests for Protective Coatings

Outline of Work:

A cooperative series of weathering tests of exterior enamels was arranged by Group II on Oleoresinous Type Enamels (C. Y. Hopkins, chairman) of Subcommittee VII to determine the degree of correlation between the effects of outdoor exposure and the results of various accelerated weathering methods.

A series of six enamels was selected and panels were made up at a central laboratory, using a finishing schedule previously agreed upon by the group. Each cooperator was supplied with one or more sets of six panels ready for exposure. Twelve members of the group carried out accelerated weathering tests, beginning on February 1, 1939. Eleven members carried out exterior exposure tests from March 1, 1939, to March 1, 1940.

Each cooperator kept a record of the condition of his own sets of panels during the tests. At the end of the exposure periods, the record sheets were collected and the results tabulated. In a few cases where complete records had not been kept, the panels were examined and rated at the end of the tests.

Materials:

The enamels were six in number and were made by pigmenting three different varnish vehicles in two colors—green and tan. The vehicles were of types in common use.

The panels were of automobile body steel of No. 22 gage. Outdoor test panels were 4 by 12 in., and the accelerated test panels were 2¾ by 5½ in. They were cleaned of all rust and grease and given a coat of gray primer, film thickness 0.0013 to 0.0020 in. After air-drying overnight, the primer was wet-sanded to an average film thickness of 0.0013 in. Two coats of enamel were applied, each coat having an average thickness of 0.001 in.

The backs of the panels were coated with the gray primer.

Methods of Exposure:

The outdoor exposures were at 45 deg. facing south, in locations in Florida, several Northern States, and Canada.

The accelerated weathering test procedures were those in regular use in the various cooperating laboratories. Six cooperators used an enclosed carbon arc as the light source, four used a test embodying a mercury arc lamp, two employed an open carbon arc and one a salt spray box. The accelerated weathering test procedures used by the cooperators are briefly described as follows:

Cooperator	Accelerated Weathering Test Procedures (Calculated to 24 hr. Basis)
1	Carbon arc (Type A). Type 70 solid carbons. Light about 21 hr. Water spray 24 hr. Water alkaline (8.1) and hardness 86 ppm. Arc voltage 145 d-c.
2	Salt spray. No light.
3	Mercury arc (Type C).
4a	Carbon arc (Type A). Solid and cored carbons. Light 15 hr. Water 15 hr. Rest 9 hr. Water alkaline (7.7) and moderately hard (90 ppm.). Arc voltage 140 d-c.

Cooperator	Accelerated Weathering Test Procedures (Calculated to 24 hr. Basis)
4b	Carbon arc (Type B). Light 12 hr. Water 12 hr. Rest 12 hr. Water alkaline (7.7) and moderately hard (90 ppm.). Arc voltage 50 d-c.
5	Carbon arc (Type A). Solid and cored carbons. Light 18 hr. Water 16 hr. Rest 4 hr. Water alkaline and soft. Arc voltage 125 d-c.
6	Mercury arc (Type C), high and low humidity. Light 12 hr. Water $\frac{3}{4}$ hr. High temperature and humidity 11 hr. Water slightly hard and slightly alkaline.
8	Carbon arc (Type A). Solid and cored carbons. Light 20 hr. with high humidity. Water spray 1 hr. Immersion in ice water 1 hr. Rest 2 hr. Water neutral and hard.
9	Carbon arc (Type A). Water neutral and soft (30 ppm.).
10	Mercury arc (Type C), high and low humidity. Light 16 hr. Water spray 2 hr. Refrigeration 2 hr. Rest period 4 hr. Water hard.
11	Mercury arc (Type C). Light only. No water spray.
12	Carbon arc (Type B). Light and water continuous. No rest period. Water hard.
13	Carbon arc (Type A). Light 22 hr. Water spray 22 hr. Heavy spray $1\frac{1}{2}$ hr. Distilled water.

Recording of Test Results:

Each cooperator made regular inspections of his exposure panels, using the record forms of the Federation of Paint and Varnish Production Clubs. Accelerated test panels were inspected weekly and outdoor panels monthly.

The effects of weathering were recorded mainly under the headings: checking, chalking, decline in gloss, change in color. At the conclusion of the tests, the record sheets and the exposed panels were examined by a small committee and the panels were rated in order of durability under each of the four headings listed above.

Analysis of Data:

The rankings based on the recorded observations of each cooperator are given in Tables I and II. The six enamels used in the tests were numbered 29 to 34, inclusive.

Comparison of Outdoor and Accelerated Test Results:

It is apparent from the data in Tables I and II that all cooperators do not agree as to the rank of the panels, either in the accelerated or the outdoor tests. Indeed, it would be surprising if complete agreement did occur, since the differences with respect to certain properties are quite small and the probability of all persons reporting identical results is remote.

Checking.—Most cooperators found early checking on panels Nos. 30 and 33 and little or no tendency for the remaining panels to check. The accelerated tests indicate that panels Nos. 30 and 33 are about equal in this respect, while the outdoor tests show more or earlier checking on panel No. 30. However, since both panels show checking in about one month outdoors, the difference is not of great consequence. It is observed that the open carbon arc tests placed these two panels in the same order as did the outdoor tests.

Chalking.—There is a good deal of variation in the ratings for chalking, partly owing to the difficulty in judging degree of chalking and partly to differences in conditions of exposure. There is little doubt about the superiority of panels Nos. 31 and 34. Panel No. 30 appears to be better than panel No. 29 in outdoor exposure, while the reverse is true in the accelerated test. It is to be noted that the variation in exposure results is as great in the outdoor as in the accelerated tests.

Gloss Retention (Before Polishing).—The gloss of the panels before polishing is not difficult to judge and fairly good agreement is shown with respect to this characteristic in the outdoor tests. The accelerated test results are less concordant, but the consensus is the same. Those using the enclosed carbon arc were in closest agreement with the outdoor tests. The mercury arc exposure placed panel No. 32 in second or third position, contrary to the results of the other tests, and was also responsible for the low ratings of panel No. 34.

TABLE I.—RANKING OF TEST PANELS IN OUTDOOR TESTS.
Ranking of Panels: 1 = Best, 6 = Poorest.

Cooperator	1	2	3	4	5	6	7	8	9	10	11	
Location	Chicago, Ill.	Detroit, Mich.	Marietta, Ohio	Washington, D. C.	Ottawa, Canada	Buffalo, N. Y.	Jacksonville, Fla.	Marcus Hook, Pa.	Bloomfield, N. J.	Palmerton, Pa.	Hialeah, Fla.	Consensus
Test Period	12 months	12 months	6 months	12 months	10 months	12 months	12 months	10 months		12 months	10½ months	
Checking Resistance[a]												
Rank												
1	..	..	..	..	..	..	..	..	..	..	..	..
2	..	..	..	..	..	..	..	..	..	..	..	..
3	..	..	..	..	..	..	..	..	..	..	..	..
4	..	..	..	..	..	34[b]	..	..	..	31	..	..
5	30, 33	33	33	33	33	33	30	33	33	33	30, 33	33
6	30, 33	30	30	30	30	30	33	30	30	30	30, 33	30
Chalking Resistance[c]												
Rank												
1	31, 34	31, 34	31	31, 34	31	31	29	31, 34	34	31	31	31
2	31, 34	31, 34	34	31, 34	34	34	31	31, 34	31	34	34	34
3	29	32	29	30	30	30	34	30	32	30	30	30
4	..	29, 33	30	32	29	32	30	29	29	29	33	29
5	..	29, 33	33	29	32, 33	29	32, 33	32, 33	30	32	29	32
6	..	30	32	33	32, 33	33	32, 33	32, 33	33	33	32	33
Gloss Retention (Before Polishing)[d]												
Rank												
1	31	31	31	31	31	31	31	31	31	31	31	31
2	34	29, 34	34	34	34	34	29	34	29, 34	29	29	34
3	29	29, 34	29	29	29	29	34	29	29, 34	34	34	29
4	32	32	30	33	30	32	30, 32	..	32	30, 32	32	32
5	30	30, 33	32	32	32, 33	30	30, 32	..	30	30, 32	30, 33	30
6	33	30, 33	33	30	32, 33	33	33	..	33	33	30, 33	33
Gloss Retention (After Polishing)[e]												
Rank												
1	..	31	[f]	[f]	31[f]	31	..	31, 34	31	31	31	31
2	..	34	..	..	32	34	..	31, 34	29, 34	29, 34	29, 34	34
3	..	32	..	..	29, 34	29	..	29	29, 34	29, 34	29, 34	29
4	..	29	..	..	29, 34	32	..	32	32	32, 33	32	32
5	..	30, 33	30, 33	30	30, 33	33	..	33	30	32, 33	30, 33	33
6	..	30, 33	30, 33	33	30, 33	30	..	30	33	30	30, 33	30

[a] Severe checking was observed on panels Nos. 30 and 33. Checking did not occur on the other four panels except in the two instances shown.

[b] Slight.

[c] Panel No. 31 proved best in resistance to chalking with panel No. 34 rated second. There was fairly general agreement with this conclusion, although exposure at Jacksonville, Fla., gave panel No. 29 the highest rating. The remaining panels were not greatly different in resistance to chalking.

[d] All cooperators found panel No. 31 to have the best gloss retention. Panels Nos. 29 and 34 were nearly as good while the remainder were very poor in this respect.

[e] Decline in gloss is less apparent after polishing and in some cases could not be judged. The ranking is the same as that for gloss retention before polishing. Panel No. 32 was improved considerably by polishing.

[f] Very little decline in gloss.

Gloss Retention (After Polishing).—Panels Nos. 30 and 33 showed poor gloss after polishing at the end of the exposure period. The other panels had suffered little decline in gloss and it was difficult

TABLE II.—RANKING OF TEST PANELS IN ACCELERATED TESTS.

Ranking of Panels: 1 = Best, 6 = Poorest.

CHECKING RESISTANCE[b]

Cooperator....	1	3	4a	4b	5	6	8	9	10	11	12	13	Consensus
Machine[a]....	Type A	Type C	Type A	Type B	Type A	Type C	Type A	Type A	Type C	Type C	Type B	Type A	
Test Period..	60 days	9 days	42 days	42 days	96 days	28 days	84 days	110 days	80 days	33 days	18 days	22 days	
Rank													
1.......	..	..	..	..	..	..	c	c	..	c	..	..	..
2.......	..	..	..	..	..	..	c	c	..	c	..	..	..
3.......	..	..	..	..	..	..	c	c	..	c	..	..	..
4.......	34	..	..	..	31[d]	..	c	c	..	c	34	34[d]	34
5.......	30	..	30, 33	33	30	33	c	c	..	c	33	30	30, 33
6.......	33	33[d]	30, 33	30	33	30	c	c	30.	c	30	33	30, 33

CHALKING RESISTANCE[e]

Cooperator..	1	4a	4b	5	6	8	9	10	11	12	13	
Machine[a]....	Type A	Type A	Type B	Type A	Type C	Type A	Type A	Type C	Type C	Type B	Type A	
Test Period..	60 days	42 days	42 days	96 days	28 days	84 days	110 days	80 days	33 days	18 days	22 days	
Rank												
1......	31	31, 34	31	31	29	31, 34	31, 34	31	31	31	31, 34	31
2......	34	31, 34	34	34	34, 30	31, 34	31, 34	..	34	34, 29	31, 34	34
3......	29	29	29, 30	29	34, 30	29	29, 30	..	30	34, 29	29	29
4......	32	32	29, 30	32	31	32	29, 30	..	29	32	30	30
5......	30	30, 33	32, 33	30	32	30	32	33	33	33	33	32
6......	33	30, 33	32, 33	33	33	33	33	30	32	30	32	33

GLOSS RETENTION (BEFORE POLISHING)[f]

Cooperator..	1	4a	4b	5	6	8	9	10	11	12	13	
Machine.....	Type A	Type A	Type B	Type A	Type C	Type A	Type A	Type C	Type C	Type B	Type A	
Test Period..	60 days	42 days	42 days	96 days	28 days	84 days	110 days	80 days	33 days	18 days	22 days	
Rank												
1......	31	31	31	31	31	31	31	31	31	31	31	31
2......	34	34	29, 34	34	29, 32	34	34	29, 32	29	29	34	34
3......	29	29	29, 34	29	29, 32	29, 30	29	29, 32	30	..	29	29
4......	32	32	30	32	30, 34	29, 30	30	30	34	..	32	32
5......	30	30	32	30	30, 34	32	32	34	32	..	30, 33	30
6......	33	33	33	33	33	33	33	33	33	..	30, 33	33

GLOSS RETENTION (AFTER POLISHING)[g]

Cooperator..	1	4a	4b	5	6	8	9	10	11	12	13	
Machine.....	Type A	Type A	Type B	Type A	Type C	Type A	Type A	Type C	Type C	Type B	Type A	
Test Period..	60 days	42 days	42 days	96 days	28 days	84 days	110 days	80 days	33 days	18 days	22 days	
Rank												
1......	31, 34	29	29, 31	h	31	30, 31	h	31	h		h	31
2......	31, 34	..	29, 31	..	29	30, 31	..	34	..	..	..	29, 34
3......	32	..	32, 34	..	32, 34	34	..	29	..	..	..	29, 34
4......	29	..	32, 34	..	32, 34	29	..	33 or 32	..	..	..	32
5......	30	30, 33	30, 33	30	33	32	..	33, 32	..	30, 33	30	30
6......	33	30, 33	30, 33	33	30	33	..	30	..	30, 33	33	33

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[a] The machines used were as follows: Type A enclosed carbon arc, Type B open carbon arc, and Type C mercury vapor lamp.

[b] While the majority of cooperators found checking on panels Nos. 30 and 33, it is evident that certain test cycles do not produce checking in conformance with outdoor weathering. Three of the mercury arc tests and two of the carbon arc tests fell in this class.

[c] No checking.

[d] Trace.

[e] The results show the superiority of panel No. 31 in resistance to chalking. Cooperator 6 reports that his test cycle s known to be unsuited to determination of chalking although it is useful in testing other characteristics.

[f] As in the outdoor tests, panel No. 31 was found to be best in gloss retention. The mercury arc tests rated panel No. 34 noticeably lower than either the carbon arc or outdoor tests.

[g] Several cooperators found such a slight decline in gloss, after polishing, that the panels could not be rated. Panels Nos. 30 and 33 were definitely the poorest, however.

[h] Very little decline in gloss.

to judge between them. Panel No. 31 is placed first in both outdoor and accelerated exposure, while panels Nos. 29, 32, and 34 are rated close to each other.

Summary of Results:

The comparative ratings are summarized in Table III. The degree of acceleration obtained in the artificial weathering tests was considered satisfactory. Some of the exposure periods shown in Table II were longer than necessary because the initial instructions were to continue the tests until checking occurred and it was later found that certain panels did not develop checking at any time.

TABLE III.—SUMMARY OF RANKINGS

Checking Resistance			Chalking Resistance		
Rank	Panel Number		Rank	Panel Number	
	Outdoor	Accelerated		Outdoor	Accelerated
1.....	[a]	[a]	1....	31	31
2.....	[a]	[a]	2....	34	34
3.....	[a]	[a]	3....	30	29
4.....	[a]	34	4....	29	30
5.....	33	30, 33	5....	32	32
6.....	30	30, 33	6....	33	33

Gloss Retention (Before Polishing)			Gloss Retention (After Polishing)		
Rank	Panel Number		Rank	Panel Number	
	Outdoor	Accelerated		Outdoor	Accelerated
1.....	31	31	1....	31	31
2.....	34	34	2....	34	29, 34
3.....	29	29	3....	29	29, 34
4.....	32	32	4 ...	32	32
5.....	30	30	5....	33	30
6.....	33	33	6....	30	33

[a] No failure by checking.

Checking.—Both tests show that panels Nos. 30 and 33 are subject to early checking and that the other panels have good resistance to checking.

Chalking.—The order of durability with respect to chalking is the same except for panels Nos. 29 and 30, which are interchanged.

Gloss (*Before Polishing*).—While this characteristic depends largely on the amount of chalk on the panel, it is easy to judge and good agreement was found between the outdoor and accelerated tests.

Gloss (*After Polishing*).—Except for the two poorest panels, the series showed only slight loss of gloss. Reasonably good agreement is observed between outdoor and accelerated tests.

Conclusions:

1. The accelerated tests identified the only two panels which fail by early checking.
2. The accelerated tests rated the panels in the proper order for resistance to chalking, except that panels Nos. 30 and 29 (rank 3 and 4) were ranked fourth and third, respectively.
3. The accelerated tests rated the panels correctly with respect to gloss retention before polishing.
4. The accelerated tests gave approximately the proper ratings for gloss after polishing. Panels Nos. 34 and 29 (rank 2 and 3) were rated as equal.
5. The agreement among cooperators' results in the accelerated tests was almost as good as among their results in the outdoor tests.
6. It is concluded that, in general, the accelerated tests predicted reasonably well the results obtained later by outdoor exposure.
7. Six persons used an enclosed carbon arc as the light source, four used a mercury vapor lamp and two an open carbon arc. Of those using the enclosed carbon arc, cooperators 1, 4a, and 13 obtained results in closest agreement with the outdoor results. Cooperator 6 was the most successful of those using the mercury arc, except in predicting chalking, where cooperator 11 was superior. Cooperator 4b was the more successful of the two using the open

carbon arc. The details of these accelerated test methods have been recorded earlier in this report.

8. It was found to be practically impossible to grade the panels with respect to color retention, either in the outdoor or accelerated test series. Accordingly, no conclusion was reached with regard to this characteristic.

Respectfully submitted on behalf of the subcommittee,

H. A. NELSON,
Chairman.

REPORT OF SUBCOMMITTEE VIII ON METHODS OF ANALYSIS OF PAINT MATERIALS

At the 1938 spring meeting of the Society, a Work Committee consisting of Messrs. A. E. Jacobsen, R. H. Sawyer, G. G. Sward, and D. L. Gamble reported to Subcommittee VIII the results of a survey of paint laboratories which indicated the No. 4 Ford consistency cup, the Gardner mobilometer, and the Stormer viscosimeter to be the most commonly used instruments for the measurement of paint consistency. At this same meeting the Work Committee was instructed to proceed with an investigation of the possibility of setting up an A.S.T.M. method of consistency measurement for gloss and enamel type paints, taking into consideration the No. 4 Ford cup, the No. 10 A.S.T.M. cup (A.S.T.M. Method D 333 – 37),[1] the Gardner mobilometer, and the Stormer viscosimeter.

The committee selected a series of five gloss or enamel type paints which were chosen to cover the range of consistency normally experienced in this type of paint. The paints were well aged and the samples distributed to the cooperating laboratories, which included the Titanium Division of National Lead Co., Krebs Pigment and Color Corp., The New Jersey Zinc Co., National Bureau of Standards, and the National Paint, Varnish and Lacquer Association Laboratory.

The results of the measurements obtained with the A.S.T.M. No. 10 cup and the No. 4 Ford cup are given in Table I. Since the Ford cup was available to only two of the laboratories, the data obtained are very meager. It is the opinion of the Work Committee that because of the sensitivity of measurements made with this type of instrument to the degree of agitation given the paint before measurement, the relatively limited range of consistencies that can be readily handled, and the necessity of close standardization, a great deal of cooperative work would have to be carried out before a satisfactory procedure could be provided. The sensitivity of the measurement to the degree of agitation given the paint prior to testing is indicated by the fact that one of the laboratories, by its procedure, was unable to measure paints D and E with either cup.

TABLE I.—RESULTS OF CONSISTENCY TESTS ON FIVE PAINTS USING A.S.T.M. AND FORD CUPS.

	Consistency, sec.				
	Paint A	Paint B	Paint C	Paint D	Paint E
A.S.T.M. Cup No. 10					
Krebs Pigment and Color Corp.	155.5	113.5	94	398.5	660
National Lead Co.	138.6	89.1	76.6	....	....
National Paint, Varnish and Lacquer Assn.	79.7	55.0	49.6	675	560
National Bureau of Standards	120	94	75	525	842
New Jersey Zinc Co.	....	....	....	....	....
Average	123.3	88.0	73.9	533	687
Maximum deviation from average, per cent	35.3	37.5	31.6	26.7	22.6
Average deviation, per cent	19.1	18.6	16.0	17.8	15.0
Ford Cup No. 4					
Krebs Pigment and Color Corp.	187	127	111	560	912
National Lead Co.	164	110.4	98.7	....	....
National Paint, Varnish and Lacquer Assn.	....	....	....	....	....
New Jersey Zinc Co.	....	....	....	....	....
Average	175.5	118.7	104.9	560	912
Average deviation, per cent	6.6	7.0	5.8	....	....

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[1] Standard Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 37), 1939 Book of A.S.T.M. Standards, Part II, p. 801.

The complete data obtained by these cooperating laboratories with the Gardner mobilometer and the Stormer viscosimeter are given in Tables II and III, respectively. In Table IV are tabulated the consistency measurements obtained on the five paints with the Stormer viscosimeter expressed in terms of load in grams required to produce a shearing rate of 100 revolutions in 30 sec. Similar data are presented for the results obtained with the Gardner mobilometer expressing the consistency in terms of load in grams required to produce a shearing rate of 10 cm. in 20 sec. These shearing rates were selected because they have been established as being in the significant range as regards the painter's judgment of consistency as determined with the spatula. In the case of the Gardner mobilometer the results are corrected to a single paint (paint C) taken as the standard and assigned a numerical

TABLE II.—RESULTS OF CONSISTENCY TESTS ON FIVE PAINTS USING GARDNER MOBILOMETER.

	Consistency, time in seconds for loads given in grams														Load in grams for 10 cm. in 20 seconds
	Stem	25	50	100	150	200	250	300	350	400	500	550	600	700	
							PAINT A								
Krebs Pigment and Color Corp.[a]	....	..	....	....	17	....	....	....	....	....	....	..	....	....	
National Lead Co.	....	..	....	53	30.5	22	16	13	10.8	....	....	..	....	....	206
National Paint, Varnish and Lacquer Assn.	....	..	....	43	26.6	19.4	15.4	12.8	10.8	9.5	....	..	....	....	193
National Bureau of Standards	....	..	....	45.5	28	22	....	13.5	....	....	....	..	....	....	215
New Jersey Zinc Co.	....	..	....	39.5	24	18	13.5	11	....	8	6	..	....	....	180
							PAINT B								
Krebs Pigment and Color Corp.[a]	19.5	17	13.5	9.5	8	7	5.5	5	....	....	....	..	....	....	93
National Lead Co.	....	..	....	22	14.2	10	7.9	....	....	....	....	..	....	....	107
National Paint, Varnish and Lacquer Assn.	....	..	....	16.2	10.1	7.3	....	....	....	....	....	..	....	....	87
National Bureau of Standards	....	..	....	25.1	14.1	10	....	6	....	....	....	..	....	....	113
New Jersey Zinc Co.	....	..	....	18	11.5	8.5	6.5	5	....	4	3	..	....	....	90
							PAINT C								
Krebs Pigment and Color Corp.[a]	22.5	18	15	11	9	7.5	6.5	6	....	....	....	..	....	....	106
National Lead Co.	....	..	....	24	14.7	11	8.2	....	....	....	....	..	....	....	115
National Paint, Varnish and Lacquer Assn.	....	..	....	24.2	15.5	11	8.9	....	....	....	....	..	....	....	119
National Bureau of Standards	....	..	....	23.2	15.1	9.9	....	6.7	....	....	....	..	....	....	113
New Jersey Zinc Co.	....	..	....	19	12	9	7	6	....	5	4	..	....	....	94
							PAINT D								
Krebs Pigment and Color Corp.[a]	....	..	....	....	47	....	....	....	....	....	....	..	....	....	....
National Lead Co.	....	..	....	....	....	54.5	....	30	....	18.6	14.0	..	....	....	380
National Paint, Varnish and Lacquer Assn.	....	..	....	....	....	45.6	....	25	....	16.5	12.4	..	9.9	....	348
National Bureau of Standards	....	..	....	300	121	61	....	31.4	....	....	....	..	....	....	....
New Jersey Zinc Co.	....	..	....	....	88	53	34	26	....	16.5	12	..	....	....	352
							PAINT E								
Krebs Pigment and Color Corp.[a]	....	..	....	....	68.5	....	....	....	....	....	....	..	....	....	....
National Lead Co.	....	..	....	....	....	80	....	51	....	34.6	24.1	..	18.6	16.1	564
National Paint, Varnish and Lacquer Assn.	....	..	....	....	....	....	....	47.2	....	31.9	23.8	..	18.9	....	578
National Bureau of Standards	....	..	....	275	136	89	....	53.5	....	....	....	..	....	....	....
New Jersey Zinc Co.	....	..	....	....	122	77	57.5	45	....	29	22	19	16.5	13.5	520

[a] Data by the Krebs laboratory is uncorrected for weight of the plunger. Add 94 g. to the recorded load in grams.

value of 100. This was done because the correction factors for reducing to terms of the master mobilometer were not available for all the instruments. In the case of the Stormer viscosimeter reduction to a common basis was not found necessary since the results obtained were in such close agreement.

TABLE III.—RESULTS OF CONSISTENCY TESTS ON FIVE PAINTS USING THE STORMER VISCOSIMETER.

	Consistency, time in seconds for load given in grams												
	100	150	196.2	200	250	275	300	400	450	500	600	700	800
PAINT A													
Krebs Pigment and Color Corp.	..	....	....	37	29	..	24	..	....	..	..	..	..
National Lead Co.	..	....	....	36	28	..	23	..	....	..	..	..	..
National Bureau of Standards	..	....	35.3	....	..	..	..	..	....	..	..	..	..
New Jersey Zinc Co.	..	....	....	35	28	26	..	..	....	..	..	..	..
PAINT B													
Krebs Pigment and Color Corp.	36	26	....	21	..	..	..	..	....	..	..	..	..
National Lead Co.	42	28	....	....	..	..	..	..	....	..	..	..	..
National Bureau of Standards	..	....	20.2	....	..	..	..	..	....	..	..	..	..
New Jersey Zinc Co.	39	27.5	....	22	..	..	..	..	....	..	..	..	..
PAINT C													
Krebs Pigment and Color Corp.	38	28	....	22	..	..	..	..	....	..	..	..	..
National Lead Co.	40	28.5	....	....	..	..	..	..	....	..	..	..	..
National Bureau of Standards	..	....	20.9	....	..	..	..	..	....	..	..	..	..
New Jersey Zinc Co.	39	28	....	22.5	..	..	..	..	....	..	..	..	..
PAINT D													
Krebs Pigment and Color Corp.	..	....	....	....	..	..	..	44	37	..	..	..	..
National Lead Co.	..	....	....	....	..	..	..	..	....	34	23	..	..
National Bureau of Standards	..	....	550	....	..	..	..	..	....	..	..	..	..
New Jersey Zinc Co.	..	....	....	....	..	..	..	50	40.5	32	24	..	..
PAINT E													
Krebs Pigment and Color Corp.	..	....	....	....	..	..	..	64	55	..	..	..	..
National Lead Co.	..	....	....	....	..	..	..	..	....	..	40	30	25
National Bureau of Standards	..	....	283	....	..	..	..	..	....	..	..	..	..
New Jersey Zinc Co.	..	....	....	....	..	..	..	..	....	..	36	30	25

TABLE IV.—RELATIVE CONSISTENCY OF PAINTS, DATA FROM STORMER AND MOBILOMETER TESTS.

	Consistency by Stormer Viscosimeter, weight in grams to produce shearing rate of 100 revolutions in 30 in.					Consistency by Gardner Mobilometer (20 in. Reading), weight in grams to produce shearing rate of 10 cm. in 20 in.				
Paint	A	B	C	D	E	A	B	C	D	E
Krebs Pigment and Color Corp.	242	122	134	526	712	....	94	100	....	....
National Lead Co.	234	136	139	532	694	179	93	100	330	490
National Paint, Varnish and Lacquer Assn.	....	....	....	....	....	162	73	100	292	485
National Bureau of Standards	....	....	....	....	....	190	100	100	....	....
New Jersey Zinc Co.	231	131	135	530	694	192	96	100	374	554
Average	236	130	136	529	700	181	91	....	332	506
Maximum deviation, per cent	2.5	6.0	2.2	0.5	1.7	10.5	20.0	....	12.6	9.5
Average deviation, per cent	1.7	3.8	1.5	0.4	1.1	5.6	8.1	....	8.4	5.5

The agreement between the laboratories is found to be best with the Stormer instrument. In the case of the Gardner mobilometer, however, it is felt that with closer standardization of the instruments and some further cooperative work it could be made to serve as a satisfactory instrument on which to base a method of consistency measurement for the type of paint under consideration.

In view of its general experience with these instruments and the good agreement of results obtained with the Stormer viscosimeter in this series of cooperative tests, the Work Committee is of the opinion that a satisfactory method of test for the consistency of gloss and enamel paints based on this apparatus can be provided at this time without further work. A Method of Test for Consistency of Enamel Type Paints is accordingly submitted herewith[2] for publication as tentative.

The committee feels that the rotational type of viscosimeter is the most universal as regards general utility of the common instruments now available and that for this reason it should be employed as the basic instrument in setting up consistency test methods. Optional methods employing the mobilometer and cups should be provided when feasible.

Respectfully submitted on behalf of the subcommittee,

E. F. HICKSON,
Chairman.

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[2] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 316.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 on Petroleum Products and Lubricants held three regular meetings during the past year (in June at Atlantic City, N. J., in January at Detroit, Mich., and in March at Washington, D. C.).

During the year the following subcommittees and technical committee have been organized: Subcommittee X on Determination of Naphtha and Chloroform Insolubles in Used Oils, Subcommittee XI on Determination of Sulfur, Chlorine, and Phosphorus in EP Lubricants, and Technical Committee C on Turbine Oils, with two sections, I on Oil Systems of Turbines, and II on Rusting.

Table I shows the changes in status during the year of American standards and American tentative standards for petroleum products, approved by the American Standards Association on the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants.

Subsequent to the 1939 annual meeting, Committee D-2 presented to the Society through Committee E-10 on Standards a revision of the Tentative Specifications for Fuel Oils (D 396 – 38 T). This revision was accepted[1] by Committee E-10 on August 24, 1939, and the specifications in their revised form appear in the 1939 Book of A.S.T.M. Standards.[2]

The voting membership of Committee D-2 now consists of 34 producers, 31

TABLE I.

Title	A.S.T.M. Designation	ASA Number
REVISIONS OF AMERICAN STANDARDS		
Test for Burning Quality of Kerosine Oils	D 187 - 39	Z11.17 - 1939
Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue)	D 189 - 39	Z11.25 - 1939
Test for Cloud and Pour Points	D 97 - 39	Z11.5 - 1939
Test for Distillation of Natural Gasoline	D 216 - 39	Z11.11 - 1939
Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer	D 287 - 39	Z11.31 - 1939
Test for Sulfur in Petroleum Oils by Bomb Method	D 129 - 39	Z11.13 - 1939
Test for Thermal Value of Fuel Oil	D 240 - 39	Z11.14 - 1939
APPROVAL OF AMERICAN TENTATIVE STANDARDS AS AMERICAN STANDARDS		
Test for Knock Characteristics of Motor Fuels	D 357 - 39	Z11.37 - 1939
Viscosity-Temperature Charts for Liquid Petroleum Products	D 341 - 39	Z11.39 - 1939
Definitions of Terms Relating to Petroleum	D 288 - 39	Z11.28 - 1939
REVISION OF AMERICAN TENTATIVE STANDARD		
Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter	D 155 - 39 T	Z11.34 - 1939
WITHDRAWAL OF AMERICAN TENTATIVE STANDARD		
Test for Expressible Oil and Moisture in Paraffin Waxes	D 308 - 29 T	Z11.27 - 1932

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee D-2 reported results of the letter ballot vote as follows: Of a total membership of 75, 57 members returned their ballots, of whom 38 voted affirmatively, 7 negatively, and 12 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part III, p. 586.

TABLE II.—DATA ON DROPPING POINT TEST.

A comparison of proposed tentative method and method published as information in 1937. Temperatures in deg. Fahr.

Laboratory	Cup Grease (Lime Base)		Fiber Grease (Soda Base)		Wheel-Bearing Grease (Soda Base)		Grease (Aluminum Base)	
	New Method	1937 Method	New Method	1937 Method	New Method	1937 Method	New Method	1937 Method
No. 1	208[a]	209	355	327	443	356[b]	241[c]	213
No. 2	210	210	351	318	442	421	234	...
No. 3	211	211	348	345	440	409	232	206
No. 4	208	210	350	342	437	443	226	204
No. 5	210	210	352	315	442	356[b]	229	215
No. 6	207	...	352	...	443	...	229	...
No. 7	210	...	356	...	...	...	235	...
No. 8	211	206	350	312	445	416	219	205
No. 9	208	211	349	348	407[b]	316	225	212
Average	209	209	351	330	442	420	230	209
Maximum deviation from average	2(3)	3	5(8)	18	5	23(26)	11(14)	6(9)
Average deviation from average	1	1	2	13	2	9	5	4

[a] The data in this tabulation represent in most cases the average of 2 to 4 tests. In cases where the "maximum deviation from the average" of individual test results not shown in the tabulation exceeds the maximum deviation based upon laboratory averages, this value is shown in parentheses.

[b] Data omitted from averages.

[c] In this tabulation there appears to be a change of dropping point with time. Tests by laboratories Nos. 2 to 7 were made about 10 days, and tests by laboratories Nos. 8 and 9 about 20 days, after the test by laboratory No. 1. A second test by laboratory No. 1 30 days after the first test gave a value of 220 F.

consumers, and 12 general interest members.

Subcommittee IV on Grease (M. B. Chittick, chairman) has proposed a new Tentative Method of Test for Dropping Point of Lubricating Grease and has submitted data as shown in Table II on the new method and on the method published as information in 1937.

The subcommittee has also proposed a new method for determining water in grease, which is to be added to the Standard Methods of Analysis of Grease (D 128 – 37). The new procedure is more convenient and rapid than the present method. Comparative data on the proposed and present methods have been submitted as shown in Table III.

TABLE III.—PERCENTAGE OF WATER IN LUBRICATING GREASE.

A comparison of proposed new method and present A.S.T.M. method

Laboratory	Sample A		Sample B		Sample C	
	Proposed Method	Present Method	Proposed Method	Present Method	Proposed Method	Present Method
No. 1	0.80	1.0	0.30	....	trace	none
No. 2	1.10	1.1	0.20	0.70	trace	none
No. 3	1.20	1.4	0.10	0.60	trace	trace
No. 4	1.12	1.0	0.10	0 30	trace	none
No 5	1.23	1.0	0.17	0.12	trace	none
No 6	1.10	1.0	0.28	0.18	trace	none
No 7	1.10	1.0	0.25	0.40	trace	none
No. 8	0.90	0.9	0.30	0.30	0.06	0.04
Average	1.07	1.05	0.21	0.37	....	....
Maximum deviation from average	0.27	0.35	0.11	0.33	....	....
Average deviation from average	0.14	0.10	0.07	0.19	....	....

Viscosity Index and Viscosity Conversion Tables.—The committee recommends that the Society arrange for the distribution of viscosity index tables[3] to avoid the necessity of computing viscosity index according to the procedure described in the new tentative method based upon the Dean and Davis system which is being presented for publication, as mentioned later in this report. The committee also recommends that the Society print detailed viscosity conversion tables[3] which will represent an extension of the short table appearing in the Standard Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (D 446 – 39).

Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman) has prepared a Proposed Method of Test for Neutralization Number of Petroleum Products and Lubricants which is published as information only and appears in Appendix I. The subcommittee has submitted the following data obtained by the use of the proposed method on used oils which do not emulsify badly or color alcohol excessively:

Laboratory	Neutralization Numbers of Used Oils		
	Sample 10	Sample 11	Sample 12
A	0.38	0.11	0.27
B	0.37	0.09	0.19
C	0.40	0.09	0.18
D	0.38	0.12	0.22
E	0.37	0.10	0.22
F	0.50	0.13	0.19
H	0.42	0.13	0.24
I	0.46	0.12	0.23
J	0.43	0.13	0.26
K	0.44	0.13	0.27
Average	0.415	0.115	0.227
Average Deviation	0.035	0.014	0.026

Also, comparative data by the proposed and present methods have been obtained on two new oils as follows:

Laboratory	Neutralization Numbers of New Oils (Alkali added in 0.1-ml. increments)			
	Sample 1		Sample 2	
	Proposed Method	Method D 188-27 T	Proposed Method	Method D 188-27 T
D	0.79	0.84	2.39	2.38
G	0.83	0.83	2.40	2.40

Subcommittee XVIII on Aniline Point (Eugene Ayres, chairman) has prepared a Proposed Method of Test for Aniline Point of Petroleum Products which is published as information only and appears in Appendix II.

Subcommittee XXIII on Carbon Residue (L. C. Beard, Jr., chairman) has recommended a revision[4] of the Tentative Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 – 39 T), substituting a metal coking bulb for the glass bulb described in the present method. The metal coking bulb has advantages over the glass bulb, as follows: It is easier to weigh the sample into the metal bulb; it can be manufactured to closer tolerances; and it is more economical to use because it can be easily cleaned and used repeatedly. The following comparative data are submitted demonstrating that the differences in results with the two types of bulbs are within the limits of reproducibility of the method:

Comparison of Metal and Glass Bulbs

Range of Carbon Residues, per cent	Number of Determinations	Difference[a] Between Metal- and Glass-Bulb Values, average, per cent
0.0 to 0.5	20	−12
0.0 to 0.5[b]	11	−2.5
0.51 to 1.00	20	+1.2
1.01 to 10.00	34	+2.1

[a] $\frac{\text{Metal-Bulb Values} - \text{Glass-Bulb Values}}{\text{Glass-Bulb Values}} \times 100$

[b] One laboratory obtained consistently lower values with the metal bulb on samples in this range of carbon residue only. The data in this line exclude the data from this laboratory.

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[3] In accordance with this recommendation, the viscosity index and viscosity conversion tables have been published separately by the Society.

[4] The revision of this method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 198.

The revision of the method also contains a provision for determining carbon residues on a 10 per cent residuum similar to that in the Standard Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 39). The following tabulation contains data for carbon residues on 10 per cent residua by both methods. There is good agreement between the data obtained with the glass and the metal bulbs:

Laboratory	Carbon Residues on 10 per cent Residua		
	Ramsbottom Carbon Residues		Conradson Carbon Residues
	Glass Bulb	Metal Bulb	
1	0.22 (6)[a]	0.20 (6)[a]	0.15 (3)[a]
2	0.21 (4)	0.18 (4)	..
3	0.23 (2)	0.22 (2)	..
4	0.24 (2)	0.21 (4)	0.14 (2)
5	0.24 (2)	0.23 (2)	0.14 (2)
Average	0.23	0.21	0.14

[a] Numbers in parentheses indicate number of determinations made by each laboratory.

Technical Committee F on Diesel Fuel Oils (C. G. A. Rosen, chairman) has prepared revisions of the Proposed Method of Test for Ignition Quality of Diesel Fuels and the Classification of Diesel Fuel Oils, which were published as information in the 1938 annual report;[5] these are again published as information only and appear in their revised form in Appendices III and IV.

I. New Tentative Standards

The committee recommends that the following three methods be accepted for publication as tentative:

Tentative Methods of:[6]

Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum), proposed by Subcommittee I on Pharmaceutical Tests (V. F. Beyer, chairman),

Test for Dropping Point of Lubricating Grease, proposed by Subcommittee IV on Grease (M. B. Chittick, chairman), and

Calculating Viscosity Index, proposed by Subcommittee V on Viscosity (J. C. Geniesse, chairman).

II. Revisions of Tentative Standards

The committee recommends revisions of five tentative standards, as follows:

Tentative Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 39 T):[7]

A revision[4] of this method as mentioned earlier in this report is recommended by Subcommittee XXIII on Carbon Residue (L. C. Beard, Jr., chairman).

Tentative Method of Test for Distillation of Plant Spray Oils (D 447 - 39 T):[8]

The following revision is recommended by Subcommittee XVII on Plant Spray Oils (J. B. Terry, chairman) to provide for the alternative use of an electric heater instead of a gas burner in carrying out the distillation:

Section 2.—Add a new Paragraph *(f)* to read as follows, relettering the subsequent paragraphs accordingly:

> *(f) Electric Heater.*—An electric heater which may be used instead of a gas burner provided it is equipped with a hard asbestos board not less than $\frac{1}{4}$ in. in thickness and 6 in. square, with a hole $2\frac{3}{4}$ in. in diameter centered over the heating element. The general characteristics of the electric heater shall be such that the results obtained by its use in distillation will duplicate those obtained with the gas burner conforming to the requirements prescribed in Paragraph *(e)*. When an electric heater is used, that portion of the shield below the asbestos plate may be omitted.

Tentative Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 39 T):[9]

The following revisions are recommended by Subcommittee XXII on

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, pp. 392, 398 (1938).

[6] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M Standards, Part III, pp. 201, 208, and 224, respectively.

[7] 1939 Book of A.S.T.M. Standards, Part III, p. 596.

[8] 1939 Book of A.S.T.M. Standards, Part III, p. 608.

[9] 1939 Book of A.S.T.M. Standards, Part III, p. 637.

Natural Gasoline (G. G. Oberfell, chairman) to clarify the description of the apparatus and to permit more rapid sampling:

Section 2 (e).—Omit the second and third sentences of this paragraph and substitute for them the following sentence:

> In determining the volume ratio, the volumes of the air and gasoline chambers shall be ascertained by carefully measuring the volume of water required to fill that portion of the chambers occupied by air or gasoline on initial assembly of the apparatus.

Section 8.—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> The chilled sampling apparatus shall consist of a bath of sufficient size to accommodate both the sample container and a cooling coil [formed of $\frac{1}{4}$-in. outside diameter, No. 20 gage copper tubing] *of about 25 ft. of copper tubing, $\frac{3}{8}$ in. or less in outside diameter.*

Tentative Method of Test for Gum Stability of Gasoline (D 525 - 39 T):[10]

The following revisions are recommended by Technical Committee A on Gasoline (C. B. Veal, chairman) to correct an error in one dimension of the bomb and to provide a more general requirement for gasket material.

Fig. 1.—Change the description, "Octagon $2\frac{3}{4}$" Across Flats" to read "Octagon $2\frac{3}{8}$" Across Flats."

Section 2 (a).—Change the second sentence from its present form: namely,

> 2. (*a*) The composition of the gasket shall be approximately 80 per cent lead, 10 per cent antimony, and 10 per cent tin.

to read as follows:

> 2. (*a*) Any suitable gasket may be used that will not react with oxygen.[a]

Tentative Specifications for Gasoline (D 439 - 37 T):[11]

The following revisions are recommended by Technical Committee A on Gasoline (C. B. Veal, chairman) to clarify the heading of Table I and to revise the octane number requirements to conform more nearly to current trade practice:

Table I.—Change the heading over the distillation temperatures to read as follows:

Minimum Percentages to be Evaporated at
Temperatures, deg. Fahr., Shown Below
10 per cent 50 per cent 90 per cent

Change octane numbers of 75, 67, and 45 to read 77, 70, and 50, respectively, wherever they occur in the table.

Change the reference to "1937" to read "1940" in footnotes "*b*" and "*d*".

III. REVISIONS OF STANDARDS

The committee recommends for immediate adoption, revisions in eight standard methods of test, as indicated below, and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 38):[12]

The following revisions are recommended by Subcommittee VIII on Distillation (E. W. Dean, chairman) to make the description of the condenser less restrictive without affecting the results and to emphasize the importance of specifying whether the distillation data are reported as volumes recovered or volumes evaporated:

Section 2 (b).—Change from its present form to read as follows:

> (*b*) *Condenser.*—A condenser (Fig. 2 shows one of the approved types) $\frac{9}{16}$ in. (14.29 mm.)

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[a] Reactivity with oxygen is determined by the following test: Place the gasket under test in the bomb in the absence of gasoline and use a gasket of similar composition in the attachment of the cover. Fill the bomb with oxygen to a pressure of 100 psi. and immerse the bomb in a bath maintained at a temperature of 211.5 ± 0.5 F. If the pressure does not drop more than 2 lb. in a 24-hr. period, the gasket may be considered nonreactive in the presence of oxygen.

[10] 1939 Book of A.S.T.M. Standards, Part III, p. 611.

[11] 1939 Book of A.S.T.M. Standards, Part III, p. 589.

[12] 1939 Book of A.S.T.M. Standards, Part III, p. 117.

in outside diameter made of No. 20 Stubbs gage seamless brass tubing, 22 in. (55.88 cm.) in length. It shall be set so that approximately 15.5 in. (39.4 cm.) of the tube will be in contact with the cooling medium contained in the condenser jacket, subsequently described, with about 2 in. outside the cooling bath at the upper end, and 4.5 in. outside the cooling bath at the lower end. The length of tube projecting at the upper end shall be straight and shall be set at an angle of 75 deg. with the vertical. The section of the tube inside the cooling bath may be either straight or bent in any suitable continuous, smooth curve. The average gradient shall be 0.259 in. per linear inch of condenser tube (sine of angle of 15 deg.) and no section of the immersed portion of the condenser tube shall have a gradient less than 0.24 in. nor more than 0.28 in. per linear inch of tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 1 to 1¼ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle. The capacity of the cooling bath shall be not less than 340 cu.in. (5.55 liters) of cooling medium. The arrangement of the tube in the cooling bath shall be such that its center line shall be not less than 1¼ in. below the plane of the top of the bath at its point of entrance and not less than ¾ in. above the floor of the bath at its exit. Clearances between the condenser tube and the walls of the bath shall be at least ½ in. except for the sections adjacent to the points of entrance and exit. Multiple installations are permissible, provided they conform to the dimensional requirements and the capacity of the bath is not less than 340 cu.in. per tube.

Section 4 (h).—Add the following sentence at the end of this paragraph:

> Clear distinction shall be made between the inclusion or exclusion of the distillation loss figure in the report of the volume of distillate obtained at any prescribed temperature. In the first case the figure shall be reported as the volume *evaporated*, in the second, as the volume *recovered*.

Standard Method of Test for Distillation of Natural Gasoline (D 216 – 39):[13]

The following revisions are recommended by Subcommittee XXII on Natural Gasoline (G. G. Oberfell, chairman) to make the description of the condenser less restrictive without affecting the results, to improve the reproducibility of the initial portion of the distillation, and to emphasize the importance of specifying whether the distillation data are reported as volumes recovered or volumes evaporated:

Section 2 (b).—Make the same change in this paragraph as is recommended above in Section 2 (*b*) of Method D 86.

Section 4 (e).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*e*) The thermometer [provided with a cork], *previously cooled to a temperature of 32 to 40 F. (0 to 4.5 C.) and dried*, shall be fitted tightly into the flask *with a suitable cork* so that it will be in the middle of the neck, and so that the lower end of the capillary tube is on a level with the inside of the bottom of the vapor outlet tube at its junction with the neck of the flask. [The thermometer shall be approximately at room temperature when placed in the flask.]

Section 5 (g).—Make the same change in this paragraph as is recommended above in Section 4 (*h*) of Method D 86.

Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 – 36):[14]

The following revision is recommended by Subcommittee XII on Flash Point (J. B. Terry, chairman):

Section 8.—Change the rate of heating in the third sentence from "not less than 4 nor more than 6 F. per min." to read "not less than 2 nor more than 3 F. per min."

This reduction in rate results in a more nearly accurate measurement of temperature and gives more reproducible results. Slightly higher flash points are obtained with the slower heating, usually in the order of 2 to 4 F.

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[13] 1939 Book of A.S.T.M. Standards, Part III, p. 127.

[14] 1939 Book of A.S.T.M. Standards, Part III, pp. 139.

Standard Methods of Analysis of Grease (D 128 – 37):[15]

The following new method of determining water is recommended by Subcommittee IV on Grease (M. B. Chittick, chairman):

Section 21.—In place of the present Section 21, which calls for a determination of water in accordance with Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 – 30), substitute the method for water as appended hereto:[16]

Standard Method of Test for Knock Characteristics of Motor Fuels (D 357 – 39):[15]

The following revisions are recommended by Technical Committee A on Gasoline (C. B. Veal, chairman) to provide a procedure for use of the method at low barometric pressures.

Section 1.—Omit the last sentence which reads as follows:

> It is applicable only at atmospheric pressures not less than 28.5 in. (724 mm.) of mercury.

Section 3.—Change the first sentence to read as follows by the addition of the italicized words:

> The knock testing unit described in this section shall be used without modification *at atmospheric pressures not less than 28.5 in. (724 mm.) of mercury; the modifications for lower atmospheric pressures are given in Appendix II.*

Change the last sentence of Paragraph (*t*) to read as follows by the addition of the italicized words and the omission of the words and figures in brackets:

> The use of a short straight-through muffler, *or equivalent*, with passage [1¼ in. in diameter] *not less than the diameter of the exhaust pipe* is permissible.

Section 6.—Omit Note 3 following Paragraph (*b*) of this section.

Change Paragraph (*g*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> (*g*) Standard Knock Intensity.—Standard knock intensity shall be that obtained with a blend by volume of 65 per cent *iso*octane and 35 per cent normal heptane under the standard operating conditions of this method *and* at a compression ratio [of 5.3 to 1 and a barometric pressure of 29.92 in. (760 mm.) of mercury (see Section 8)] *obtained by calculation in accordance with the formula given in Section 8.*

Change Paragraph (*p*) to read as follows by the addition of the italicized words:

> (*p*) Cooling Liquid.—Distilled water or rain water *shall be used to maintain jacket temperature when barometric pressure is sufficient to obtain standard temperature. At lower barometric pressures add sufficient ethylene glycol to water to obtain standard jacket temperature.*

Sections 8 and 10.—At the end of the formulas and preceding Note 7 in Section 8, and preceding Section 10 (*c*), add a new paragraph to read as follows:

> For barometric pressures less than 28.5 in. (724 mm.) of mercury substitute the basic barometric pressure given in Appendix II corresponding to the venturi size for the value of 29.92 in the formula.

Omit Note 8 following Section 8.

Appendix.—Designate the present appendix as Appendix I and add a new Appendix II to read as follows:

APPENDIX II

For engines which operate at high altitudes, it is necessary to modify the test conditions to compensate for changes in atmospheric pressure. Where the atmospheric pressure is less than 28.5 in. (724 mm.) of mercury, the following modifications shall be made:

Carburetor.—The size of the venturi (Section 3 (*o*)) shall be governed by the altitude (see Table I).

Throttle Plate.—The throttle plate (Section 3 (*p*)) shall be removed.

Standard Knock Intensity and Check of Test Conditions.—In determining the standard knock intensity (Section 8) and in the checking of test conditions (Section 10 (*b*)) the corresponding barometric pressures obtained from Table I shall be substituted for the value of 29.92 in. (760 mm.) of mercury.

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[15] 1939 Book of A.S.T.M. Standards, Part III, pp. 159, 170.

[16] This revision was adopted as standard by the Society and has been incorporated in the methods which appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 53.

TABLE I.

Nearest Altitude, ft.	Venturi Size, Diameter, in.	Basic Barometric Pressure, Inches of Mercury
1300	9/16[a]	29.92
1900	9/16	27.9
2600	19/32	27.2
3300	10/16	26.5
3900	11/16	25.9
4400	12/16	25.4
4800	13/16	25.0
5400	14/16	24.5
6000	16/16	24.0

[a] At this altitude and venturi size diameter the throttle plate prescribed in Section 3 (*p*) of this method shall be in place.

Standard Method of Test for Precipitation Number of Lubricating Oils (D 91 - 35)[17] *and*

Standard Method of Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 - 35):[17]

The following revisions are recommended by Subcommittee XIV on Water and Sediment (H. R. Gundlach, chairman) to improve the calibration of the cone-shaped centrifuge tube employed in these two methods:

Centrifuge Tubes.—Change the calibration requirements in the range 1 to 3 ml. to read as follows by the addition of the italicized requirements and the omission of those in brackets:

Range, ml.	Scale Division, ml.	Limit of Error, ml.	Numbered, ml.
[1 to 3	0.1	0.1	2, 3]
1 to 2	*0.1*	*0.1*	*2*
2 to 3	*0.2*	*0.1*	*3*

Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 30):[18]

Two revisions in this method are also recommended by Subcommittee XIV. A change in the scope of the method is submitted because of the proposal to employ a different method for determining water in grease. A revision in the calibration of the centrifuge tube is also recommended to bring the method into conformity with a change in the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) as proposed by Committee D-8 on Bituminous Waterproofing and Roofing Materials. This latter revision is being recommended jointly with Committee D-4 on Road and Paving Materials.

Section 1.—Add a new note to this section to read as follows:

NOTE.—For the determination of water in grease reference should be made to the procedure described in the Standard Methods of Analysis of Grease (A.S.T.M. Designation: D 128) of the American Society for Testing Materials.[15]

Section 5 (a).—In the note following this section change the last sentence to read as follows, by the addition of the italicized words and figures and the omission of those in brackets:

This tube shall be graduated from 0 to [25 ml.] *2 ml.* in 0.1-ml. divisions *and from 2 to 25 ml. in 0.2-ml. divisions* (see Fig. 3 (*c*) of the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials).

The committee recommends a tentative revision of the following standard method:

Standard Method of Test for Distillation of Crude Petroleum (D 285 - 36):[19]

Subcommittee XXI on Crude Petroleum (A. J. Kraemer, chairman) recommends a tentative revision of this method as follows:

Special Procedure.—Introduce a new Section 8 to read as follows:

8. *Special Procedure for Obtaining Large Volumes of Distillate.*—If it is necessary to obtain quantities of distillate large enough for octane ratings or other special tests, the following procedure may be used: A charge of crude oil shall be distilled in equipment of suitable size to provide the desired amount of naphtha distillate in a single run or in a small number of runs. The equipment shall be so designed and operated that it duplicates both the yield and

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[17] 1939 Book of A.S.T.M. Standards, Part III, pp. 187, 233.

[18] 1939 Book of A.S.T.M. Standards, Part III, p. 230.

[19] 1939 Book of A.S.T.M. Standards, Part III, p. 112.

the quality of the distillate obtained by use of the regular procedure with a 300-ml. charge. The following criteria shall be considered in deciding whether the two procedures give equivalent results:

(*a*) Yields of naphtha distillate shall not differ by more than 1 per cent.

(*b*) The distillation curves of the naphtha fractions shall not differ at any point by more than 2.0 per cent.

TABLE IV.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum)	41	0	21
Test for Dropping Point of Lubricating Grease	50	2	12
Method of Calculating Viscosity Index	55	1	8
II. REVISIONS OF TENTATIVE STANDARDS			
Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 39 T)	61	0	1
Test for Distillation of Plant Spray Oils (D 447 - 39 T)	49	0	15
Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 39 T)	56	1	7
Test for Gum Stability of Gasoline (D 525 - 39 T)	51	0	12
Spec. for Gasoline (D 439 - 37 T)	53	0	9
III. REVISIONS OF STANDARDS			
Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 38), immediate adoption	60	0	4
Test for Distillation of Natural Gasoline (D 216 - 39), immediate adoption	50	0	14
Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 36), immediate adoption	59	0	5
Analysis of Grease (D 128 - 37), immediate adoption	48	2	14
Test for Knock Characteristics of Motor Fuels (D 357 - 39), immediate adoption	48	0	13
Test for Precipitation Number of Lubricating Oils (D 91 - 35), immediate adoption	54	1	7
Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 - 35), immediate adoption	57	1	4
Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 30), immediate adoption	58	0	4
Test for Distillation of Crude Petroleum (D 285 - 36)	52	1	9
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Unsulfonated Residue of Plant Spray Oils (D 483 - 38 T), as revised	40	0	24
Spec. for Stoddard Solvent (D 484 - 38 T), as revised	52	0	12

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following two tentative standards be approved, as revised, for reference to letter ballot of the Society for adoption as standard:

Tentative Method of Test for Unsulfonated Residue of Plant Spray Oils (D 483 - 38 T),[20] on the recommendation of Subcommittee XVII on Plant Spray Oils (J. B. Terry, chairman), slightly revised as follows:

Section 3 (f).—Change the first sentence to read as follows by the addition of the italicized word and figure and the omission of the figure in brackets:

> A *suitable* centrifuge capable of whirling two test bottles filled with acid at a speed of [1000] *500* rpm.

Section 4.—In the second sentence of Paragraph (*f*) change the speed of whirling from "1000 rpm." to read "500 rpm."

Change the second sentence of Paragraph (*g*) to read as follows by the addition of the italicized figure and the omission of the figure in brackets:

> The value so obtained is the percentage of unsulfonated residue, and shall be reported to the nearest [0.1] *0.2* per cent.

Tentative Specifications for Stoddard Solvent (D 484 - 38 T),[21] on the recommendation of Technical Committee D on Stoddard Solvent (L. E. Jackson, chairman), slightly revised as follows:

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[20] 1939 Book of A.S.T.M. Standards, Part III, p. 634.

[21] 1939 Book of A. S. T. M. Standards, Part III, p. 592.

Section 4 (c).—Add a new sentence after the second sentence of the paragraph on procedure to read as follows:

> Observe within a 2-min. period.

Section 4 (d).—Change the third sentence of the paragraph on procedure to read as follows by the addition of the italicized words and the omission of those in brackets:

> Add from a graduate 20 ml. of c.p. sulfuric acid [(sp. gr. 1.83 to 1.84 at 20 C; approximately 93.2 per cent)] (*93.2 ± 0.3 per cent concentration by titration*) *previously cooled in ice water for 5 min.*

In the sixth sentence of the paragraph on procedure change the shaking time specified from "1 min." to read "60 ± 5 sec."

Following the seventh sentence of the paragraph on procedure add a new sentence to read as follows:

> A centrifuge may be used when it is desired to avoid the loss of time required for standing overnight.

V. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The following tentative standards have remained in the tentative status for two years or longer without revision:

Tentative Methods of:

Test for Ash Content of Petroleum Oils (D 482 - 38 T),

Test for Consistency of Lubricating Greases and Petrolatum (D 217 - 38 T),

Test for Sediment in Fuel Oil by Extraction (D 473 - 38 T), and

Test for Sulfur in Petroleum Oils by Lamp Method (D 90 - 34 T).

It is recommended that these tentative standards be continued in the tentative status because further work on them is in progress or in prospect.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 77 voting members, with the results shown in Table IV.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 77 voting members; 64 members returned their ballots, of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. A. BOYD,
Chairman.

R. P. ANDERSON,
Secretary.

APPENDIX I

PROPOSED METHOD OF TEST FOR NEUTRALIZATION NUMBER OF PETROLEUM PRODUCTS AND LUBRICANTS[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These methods of test are designed to indicate in petroleum products and lubricants and compounded products the presence of organic constituents having acid characteristics, and the contamination by alkalies and mineral acids.

Neutralization Number

2. (*a*) *Neutralization Number.*—Neutralization number is the number of milligrams of potassium hydroxide required to neutralize one gram of sample.

NOTE.—Neutralization number expresses the total amount of the mineral acid, and of the organic constituents having acid characteristics.

(*b*) *Alkali Neutralization Number.*—Alkali neutralization number is defined as the weight of acid required to neutralize one gram of sample, expressed in equivalent milligrams of potassium hydroxide.

(*c*) *Mineral Acid Neutralization Number.*—Mineral acid neutralization number is the number of milligrams of potassium hydroxide required to neutralize the mineral acid content in one gram of sample.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Erlenmeyer Flask.*—An Erlenmeyer flask of 250-ml. capacity, made of heat-resistant glass.

(*b*) *Burette.*—A burette of any convenient capacity (5 ml. or greater) graduated in not greater than 0.1-ml. subdivisions.

(*c*) *Balance.*—A platform balance accurate to at least 0.1 g.

(*d*) *Hot Plate.*—A hot plate capable of heating samples from room temperature to boiling within 5 min.

Special Solutions Required

4. (*a*) *Aqueous Potassium Hydroxide* (1 ml. = 5 mg. of KOH).—Dissolve 5.1 g. of c.p. potassium hydroxide in 1 liter of freshly boiled and cooled distilled water. Add a very small amount of barium hydroxide, sufficient to precipitate any potassium carbonate present. Standardize this alkali solution against

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] This proposed method represents a revision that is under consideration by the committee of the Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (D 188-27T), 1939 Book of A.S.T.M. Standards, Part III, p. 617.

a solution of benzoic acid (prepared from National Bureau of Standards' standard sample 39*e*) in the alcohol described in Paragraph (*c*), using phenolphthalein as the indicator. Titrate without heating. Correct the titration by running a blank on the same amount of alcohol, containing no benzoic acid. Adjust the potassium hydroxide solution so that 1 ml. contains 5 mg. of KOH (equivalent to 10.88 mg. of benzoic acid).

NOTE.—Fit the solution bottle with a guard tube of soda lime to prevent access of carbon dioxide. The solution should be restandardized at necessary intervals.

(*b*) *Diluted Sulfuric Acid* (1 ml. = 5 mg. of KOH).—Adjust a c.p. sulfuric acid solution so that 1 ml. of the acid will be required to neutralize 1 ml. of the standard alkali, using phenolphthalein as indicator, and titrating in a boiling solution.

(*c*) *Neutralized Alcohol Solution.*—Prepare this solution by using c.p. 95 per cent ethanol to which has been added 10 per cent by volume of methanol (Note). Make a mixture using one volume of the alcohol diluted to two volumes with distilled water, and bring to the boiling point. Remove from the hot plate, and carefully neutralize while hot with the aqueous potassium hydroxide, using 1 ml. of phenolphthalein as the indicator.

NOTE.—This composition is available under the name of: "U. S. Treasury Dept. Specially Denatured Formula 30 (Regulation No. 3—1938)." Formula 3A plus 5 per cent methanol is an equivalent.

(*d*) *Phenolphthalein Indicator.*—Dissolve 10 g. of the indicator in 1 liter of either 95 per cent pure ethanol, or 95 per cent ethanol to which 10 per cent by volume of methanol has been added.

(*e*) *Methyl Orange Indicator.*—Dissolve 1 g. of the indicator in 1 liter of distilled water. Use 0.1 ml. of this strength for titration.

Neutralization Number

5. (*a*) *Procedure.*—Weigh 20 ± 0.1 g. of a representative sample into an Erlenmeyer flask. Add 100 ml. of the neutralized alcohol solution and 1 ml. of the phenolphthalein indicator. Agitate the mixture by vigorous swirling in the unstoppered flask, place on the hot plate, and heat to boiling. Agitate by swirling during the heating process at intervals of 30 sec., or more frequently if necessary, to prevent bumping. When boiling begins, remove the flask from the hot plate and agitate the mixture by vigorous swirling for a period of 1 min. Then introduce 0.1 ml. of the KOH solution, agitate by vigorous swirling for 5 sec. (or longer if necessary to obtain thorough mixing as indicated by the absence of red streaks in the alcohol layer), and let the flask stand on an insulating base (such as sheet asbestos) until the sample and alcohol have separated sufficiently to permit a clear observation of the color of the alcohol layer. The color of this layer may best be observed by raising the flask and tipping at an angle. For satisfactory observations, good daylight or a daylight lamp should be employed. Do not allow the flask to stand between agitation and the next addition of alkali longer than is required for observation of the alcohol layer. If this layer is not pink, continue the titration as rapidly as possible by the addition of 0.1-ml. portions of the alkali, agitating and settling as described above until a pink color is observed. Then reheat the mixture to boiling, and agitate by swirling for an additional 30 sec If the color persists, the total amount of alkali added represents the amount required to neutralize the acid contained in 20 g. of the sample. If the color disappears, add alkali in 0.1-ml. portions (Note), agitating in each case for 5 sec.

(or longer if necessary) as before. When the faint pink color is again obtained, heat the sample to boiling, and swirl for 30 sec. Repeat the above procedure until the pink coloration persists after reheating and swirling for 30 sec. It is important to make a careful observation of any color change in the alcohol layer as the faint pink color may be masked by color from oil dissolved in the alcohol. In all cases, carry the titration a little beyond the first pink coloration to ascertain whether the true end point has been reached. If the addition of a small amount of alkali increases the intensity of the pink color, the point at which the color change was observed represents the end point, and the quantity of alkali added to the solution to bring about this first change or faint pink color shall be recorded as the number of milliliters of alkali required to neutralize 20 g. of the sample.

NOTE.—If a new oil is being titrated, and if the neutralization number is expected to be 0.10 or lower, a more precise value may be obtained by adding the alkali required for titration in 0.05-ml. portions.

(*b*) *Calculation.*—Calculate the neutralization number of the sample as follows:

$$\text{Neutralization number} = \frac{\text{milliliters of KOH} \times 5}{\text{weight of sample}}$$

Alkali or Mineral Acid Neutralization Number[3]

6. (*a*) *Procedure.*—Introduce 25 to 50 ± 0.1 g. of a representative sample into a 250-ml. separatory funnel, and add 100 ml. of boiling distilled water. Shake vigorously and, after sample and water have separated, drain the water layer into a 500-ml. casserole. Wash the sample in the separatory funnel twice by vigorously shaking with 50-ml. portions of boiling distilled water and, after separation, drain into the casserole. To the accumulated 200 ml. of water add 1 drop of phenolphthalein solution and bring to boiling. If solution turns pink, add 1 ml. of the indicator and titrate with the standard diluted sulfuric acid until the solution is colorless.

If the addition of 1 drop of phenolphthalein causes no change in color, add 0.1 ml. of methyl orange. If color changes to red or pink, titrate with the KOH solution until the red color is just discharged.

Titrate an equal volume of distilled water with the standard solution used, and for the blank correction subtract from the titration the amount of alkali or acid required.

(*b*) *Calculation.*—If the acid solution was required to discharge the phenolphthalein end point, calculate the alkali neutralization number as follows:

$$\text{Alkali neutralization number} = \frac{\text{milliliters of acid} \times 5}{\text{weight of sample}}$$

If the alkali solution was required to discharge the methyl orange end point, calculate the mineral acid neutralization number as follows:

$$\text{Mineral acid neutralization number} = \frac{\text{milliliters of alkali} \times 5}{\text{weight of sample}}$$

Reproducibility of Results

7. (*a*) With proper care, and strict attention to details, duplicate determinations by the same operator should not differ by more than the amounts given in the following tabulation:

Material Tested	Permissible Difference
New oils (using 0.1-ml. increments)	0.03
New oils (using 0.05-ml. increments)	0.015
Used oils (which do not form stable emulsions or do not color the alcohol layer excessively)	0.05

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[3] When the oil is acid, the acidity shall not be reported as sulfuric acid or hydrochloric acid unless the presence of these acids shall have been confirmed by tests.

NOTE.—No specific permissible differences can be given for oils which form stable emulsions or color the alcohol layer excessively.

(*b*) Results obtained by different operators, may differ by double the amounts given in Paragraph (*a*).

APPENDIX II

PROPOSED METHOD OF TEST FOR ANILINE POINT OF PETROLEUM PRODUCTS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method of test is intended for determining the aniline point of petroleum products not darker than No. 8 A.S.T.M. color,[2] provided the aniline point lies between the initial boiling point and the solidification point of the aniline - sample mixture.

Definition

2. *Aniline Point*.—Aniline point is the minimum equilibrium solution temperature for equal volumes of aniline and petroleum product.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Test Tube*.—A test tube approximately 25 mm. in diameter and 150 mm. in length, made of heat-resistant glass.

(*b*) *Jacket*.—A jacket approximately 38 mm. in diameter and 175 mm. in length made of heat-resistant glass.

(*c*) *Stirrer*.—A variable-speed, mechanical stirrer, fitted with an auger-type glass tip as shown in Fig. 1.

(*d*) *Thermometers*.—Suitable thermometers conforming to the following requirements:

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISIONS:
- −38 to +42 C., in 0.2 C.
- 25 to 105 C. in 0.2 C.
- 90 to 170 C. in 0.2 C.

TOTAL LENGTH: 403 to 409 mm. (15.87 to 16.10 in.).

STEM: Plain front, enamel back, suitable thermometer tubing.
Diameter, 6.0 to 7.0 mm. (0.236 to 0.275 in.).

BULB: Corning normal or equally suitable thermometric glass.
Length, 10.0 to 20.0 mm. (0.39 to 0.78 in.).
Diameter, 5.0 to 6.0 mm. (0.197 to 0.236 in.).

DISTANCE TO LOWEST GRADUATION LINE FROM BOTTOM OF BULB: 101 to 115 mm. (3.976 to 4.527 in.).

DISTANCE TO HIGHEST GRADUATION LINE FROM TOP OF THERMOMETER: 25 to 45 mm. (0.98 to 1.77 in.).

FILLING ABOVE LIQUID: Nitrogen gas.

TOP FINISH: Glass ring.

GRADUATION: All lines, figures, and letters clear cut and distinct. The first and each succeeding 1 C. line to be longer than the remaining lines. Graduations to be numbered at each multiple of 2 C.

IMMERSION: 51 mm. or 2.008 in.

SCALE ERROR: The error at any point of the scale when the thermometer is standardized as provided below shall not exceed 0.2 C.

STANDARDIZATION: The thermometer shall be standardized for 51-mm. immersion at standardization temperatures to be chosen and at determined stem temperatures.

CASE: The thermometer shall be supplied in a suitable case.

NOTE.—For the purpose of interpreting these specifications the following descriptions of terms apply:

The total length is the over-all length of the finished instrument.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155) of the American Society for Testing Materials, see 1939 Book of A.S.T.M. Standards, Part III, p. 598.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

(*e*) *Pipettes.*—Two 10-ml. pipettes, one provided with a suitable rubber bulb for handling aniline.[3]

(*f*) *Balance.*—A laboratory balance sensitive to 0.01 g. suitable for weighing test tube and sample when the sample cannot conveniently be pipetted.

(*g*) *Heating Bath.*—A suitable nonaqueous, nonvolatile, transparent liquid bath. An open flame or a hot-air gun may be substituted for the liquid bath, but hot water should not be used as aniline is hygroscopic.

Reagents

4. (*a*) *Aniline.*—Dry c.p. aniline over KOH pellets, decant, and distill fresh on the day of use, discarding the first and last 10 per cent. Aniline may be checked against suitable secondary standards, such as *n*-heptane or paraffinic fractions of known aniline point.

(*b*) *Anhydrous Sodium Sulfate.*

Preparation of Sample

5. The sample to be tested shall be dried by shaking for at least 2 min. with anhydrous Na_2SO_4. In the case of viscous samples, it may be necessary to remove the drying agent by filtering through a fritted (sintered) glass filter of medium porosity.

Procedure

6. (*a*) Clean and dry the apparatus. Pipette 10 ml. of aniline[3] and 10 ml. of the sample into the jacketed test tube fitted with stirrer and thermometer. If the material is too viscous for pipetting, weigh a quantity of the sample corresponding to 10 ml. at room temperature in the test tube to the nearest 0.01 g. Place the thermometer in the test tube at the proper immersion, making sure that the bulb does not touch the side of the tube.

(*b*) Stir the mixture rapidly, avoiding the inclusion of air bubbles, and heat until complete miscibility is obtained. Continue stirring, and allow the mixture to cool slowly at a rate not exceeding 1 C. per min. (Note). Turbidity may occur due to separation of addition agents which are not strictly petroleum oil. The temperature at which such turbidity occurs should be disregarded. Record the temperature at which the mixture becomes cloudy throughout with complete separation of aniline and oil. This will be the minimum critical solution temperature and not the temperature of separation of minor portions of the sample.

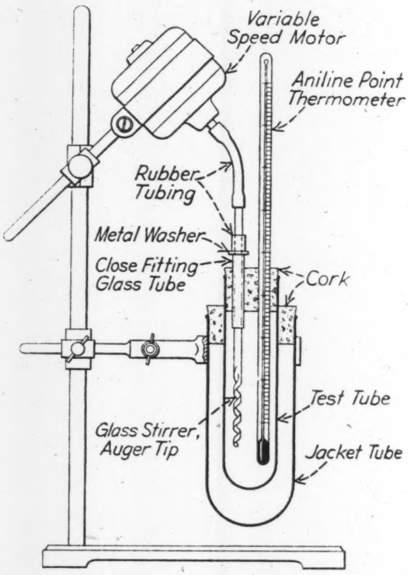

FIG. 1.—Apparatus for Aniline Point Test.

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[3] Aniline should not be pipetted directly because of its extreme toxicity. Aniline is also toxic by absorption through the skin, even in *very small quantities*, and should be handled with great caution.

(*c*) Repeat the heating and cooling operations and again record the temperature at which separation of aniline and sample occurs. If constant value is not obtained after five determinations, repeat the test using fresh quantities of aniline and sample in clean, dry equipment.

NOTE.—If the temperature at which turbidity appears is below that of the atmosphere, substitute a nonaqueous cooling medium for the heating medium.

Report

7. Report the minimum critical solution temperature of the mixture of equal volumes of aniline and petroleum product as the aniline point of the sample.

Reproducibility of Results

8. With proper care and attention to details, duplicate results should not differ by more than plus or minus 0.2 C. from the average.

APPENDIX III

PROPOSED METHOD OF TEST FOR IGNITION QUALITY OF DIESEL FUELS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method of test is intended for determining the ignition quality of Diesel fuels in terms of an arbitrary scale of A.S.T.M. cetane numbers.

A.S.T.M. Cetane Number

2. The A.S.T.M. cetane number of a Diesel fuel is defined by and is numerically equal to the percentage by volume of cetane in a mixture of cetane and α-methylnaphthalene which the fuel matches in ignition quality when compared by the procedure prescribed in this method. Thus, by definition, α-methylnaphthalene has a cetane-number rating of zero, and cetane, of 100. Cetane number is reported to the nearest whole number.

APPARATUS

Apparatus

3. The ignition quality testing unit described in this section shall be used without modification. It shall be known as the "C.F.R. Diesel Fuel Testing Unit."[2] The apparatus shall consist of a continuously-variable-compression engine, together with instrument panel, base, and suitable loading and accessory equipment, as follows:

(*a*) *Engine.*—Continuously-variable-compression, one-cylinder, with dimensions[a] as follows:

Bore, in.	3.25
Stroke, in.	4.50
Displacement, cu. in.	37.4
Turbulence combustion-chamber:	
bore, in.	1.625
length, in., adjustable	0.375 to 2.750
Valve-port diameter, in.	1.187
Connecting-rod bearing:	
diameter, in.	2.25
length, in.	1.625
Front main bearing:	
diameter, in.	2.25
length, in.	2.00
Rear main bearing:	
diameter, in.	2.25
length, in.	4.25
Piston-pin, floating, diameter, in.	1.25
Connecting-rod, length, center to center, in.	10.00
Timing-gear face, in.	1.00
Piston rings, number	5
Exhaust pipe, diameter, in.	1.25
Weight of engine, lb. (approximate)	475
Weight of complete unit, lb. (approximate)	1375

[a] All dimensions are nominal and subject to manufacturing tolerances.

(*b*) *Crankshaft.*—Fully machined, heat-treated, and counterbalanced.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

This method originated in the Automotive Diesel Fuels Division of the Cooperative Fuel Research Committee, which is composed of representatives of the American Petroleum Institute, the Automobile Manufacturers Association, the Society of Automotive Engineers, Inc., and the National Bureau of Standards.

[2] At present the sole authorized manufacturer of the C.F.R. Diesel Fuel Testing Unit is the Waukesha Motor Co., Waukesha, Wis. Other manufacturers may be approved in the future, but testing laboratories should not purchase testing units, except from the Waukesha Motor Co., without ascertaining whether such units have been approved. Inquiries in this connection should be directed to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 50 West Fiftieth Street, New York, N. Y.

(*c*) *Crankcase.*—Cast iron.

(*d*) *Connecting Rod.*—Rifle-drilled, S.A.E. No. 1045 steel, heat-treated, bearing alloy cast directly into big end.

(*e*) *Main Bearings.*—Renewable sleeve bushings, babbit-lined.

(*f*) *Valves.*—Silcrome or equivalent.

(*g*) *Camshaft:*

(*1*) *Valve lift.*—0.238 in.

(*2*) *Valve timing.*—Intake valve opens 10 deg. a.t.d.c., closes 34 deg. a.b.d.c. Exhaust valve opens 40 deg. b.b.d.c., closes 15 deg. a.t.d.c.

(*3*) *Valve clearance.*—0.010 in. clearance on both valves for checking valve lift and valve timing. (For running clearance see Section 6 (*g*).)

(*h*) *Push Rods.*—Mushroom type with lock-nut adjustment.

(*i*) *Cylinder.*—Cast iron alloy, bored and honed, Brinell hardness 220 ± 20.

(*j*) *Cylinder Head.*—Detachable variable-compression high-turbulence.

(*k*) *Cooling System.*—Evaporative.

(*l*) *Lubrication.*—Pressure feed to main, connecting-rod, piston-pin, and camshaft bearings, to idler-gear stud and gears, and to valve rocker arms.

(*m*) *Oil Heater and Thermometer.*—Electric heater in base to bring oil to operating temperature quickly, and a thermometer on the instrument panel to indicate when equilibrium temperature has been reached.

(*n*) *Air Heater and Thermometer.*—Electric heater mounted on intake silencer to maintain constant air-inlet temperature, and a thermometer at base of silencer to indicate this temperature.

(*o*) *Water Heater and Thermometer.*—Electric heater in cooling-water line between outlet of condenser and inlet to cylinder jacket to bring water to operating temperature quickly, and a thermometer between condenser and heater to indicate this temperature.

(*p*) *Injection System.*—This system shall consist of the following parts as furnished with the engine:

(*1*) Injector pump, Bosch specification PE1B50A 302.

(*2*) Injector, Bosch specification DN - 30S3.

(*3*) Fuel line (tank to pump), $\frac{3}{8}$-in. copper tubing, bottom of fuel tank to be 25 ± 1 in. above pump inlet.

(*4*) Injection line, $\frac{1}{4}$ in. in outside diameter, $\frac{1}{16}$ in. in inside diameter, and 27 in. in length.

(*5*) Vertical open riser with measuring burette located on pump sump outlet.

(*q*) *Instrumentation.*—The ignition quality is measured by the coincident-flash fixed-delay method using neon lights which are located on the periphery of the flywheel and are operated by contact points on the combustion and injection indicators. The necessary instrumentation consists of the following parts:

(*1*) The combustion indicator which mounts in a tapped hole ($\frac{7}{8}$ in. by 18 threads per inch) in the combustion-chamber and is equipped with a spring-steel indicator diaphragm 0.543 ± 0.003 in. in diameter and 0.015 ± 0.0005 in. in thickness. The contact points are mounted on bronze leaf springs 0.021 ± 0.0005 in. in thickness. Graduated spring-tension adjusting screws are provided. In addition, a graduated gap-adjusting screw is provided with a spring cushion plunger to protect the contact points against overloading as the result of combustion pressures.

(*2*) The injection indicator, which utilizes contact-point assembly parts similar to the combustion indicator, with brackets for attachment to permit operation of the contact points by movement of the injector valve.

(*3*) The neon-light mount which carries two $\frac{1}{4}$-watt, 110-v., neon bulbs located behind an aperture plate with 13 deg. spacing between openings.

(*4*) The sight tube and top dead-center reference mark with brackets.

(*5*) Flywheel slip rings and brush holders with necessary brackets and wiring for the neon-light support.

(*6*) A 110-v. d-c. generator to supply current for the injection and combustion indicators. This generator is belt driven from the power-absorbing unit described under Paragraph (*r*). The voltage is controlled to 115 ± 5 v. by means of a field rheostat located on the instrument panel.

(*r*) *Power-Absorbing Unit.*—The engine is connected by V-type belts to an electric generator which preferably should be an induction motor with synchronous characteristics, but may be any electric generator capable of maintaining proper operating conditions. In most cases the electric generator will act as a starting motor to crank the engine, but if a direct-current generator is used and no outside source of current is available, the engine may have to be cranked by hand.

(*s*) *Panel.*—A control panel with starter switches, heater switches, and oil-temperature and pressure gages is included as part of the unit.

(*t*) *Base.*—The complete unit is mounted on a cast iron base.

Foundation

4. The foundation shall be of concrete, at least 15 in. in height above the floor level, resting, if possible, directly on the ground. Otherwise, special provision must be made to eliminate vibrations that may influence ratings, and the manufacturer should be consulted. For convenience in operation, the foundation should be located at least 2 ft. from any wall. Dimensions are furnished with the apparatus.

Reference Fuels

5. The primary reference fuels[3] shall be normal cetane and α-methylnaphthalene. Secondary reference fuels,[4] suitably calibrated against these primary reference fuels, are normally used in actual testing.

Standard Operating Conditions

6. The engine shall be run under the following standard conditions:

(*a*) *Engine Speed.*—Constant, 900 ± 9 r.p.m.

(*b*) *Jacket Temperature.*—Constant within plus or minus 1 F. (0.6 C.), and at a temperature between the limits of 209 and 215 F. (98 and 102 C.).

(*c*) *Cooling Liquid.*—Distilled water or rain water, or ethylene-glycol solution when necessary at high altitude.

(*d*) *Crankcase Lubricating Oil.*—S.A.E. 30.

NOTE.—The viscosity range of crankcase lubricating oil, S.A.E. 30, is from 185 to 255 sec. Saybolt Universal at 130 F. (54.4 C.), determined in accordance with the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88) of the American Society for Testing Materials.[5]

(*e*) *Oil Pressure.*—25 to 30 psi. under operating conditions.

(*f*) *Oil Temperature.*—The electric oil heater to be used only to raise the oil temperature rapidly to the equilibrium operating temperature, 120 to 150 F. (49 to 65 C.), normal operation maintaining the equilibrium thereafter. By "equilibrium" is meant that steady temperature attained by the oil upon continuous operation of the engine without the use of the heater.

(*g*) *Valve Clearances, Cold.*—Intake 0.008 in., exhaust 0.010 in.

(*h*) *Injection Advance.*—13 deg. b.t.d.c. for rating, and 5 ± 2 deg. b.t.d.c. for combustion contact setting.

(*i*) *Injection Pressure* (injector opening pressure).—1500 ± 50 psi. (106 ± 4 kg. per sq. cm.).

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[3] Normal cetane is available from the E. I. du Pont de Nemours and Co., Inc., Wilmington, Del., and α-methylnaphthalene, from the Reilly Tar and Chemical Co., Indianapolis, Ind.

[4] High-cetane secondary reference fuel known as "Shell high-cetane reference fuel" is available from the Shell Oil Co., Inc., Wood River, Ill. Low-cetane secondary reference fuel, a mixture of α- and β-methylnaphthalenes, is available from the Reilly Tar and Chemical Co., Indianapolis, Ind.

[5] 1939 Book of A.S.T.M. Standards, Parts II and III.

NOTE: ***Injection Pressure Setting.***—The injection pump cover shall be removed and, with a suitable injection-line pressure gage connected and the injector arranged to spray into the air, the pump plunger shall be operated with a screw driver used as a lever. The pressure on the injector spring shall be adjusted until fuel spray occurs at a gage pressure of 1500 psi.

(*j*) *Injection Quantity.*—13.0 ± 0.5 ml. per min., to be checked on each test sample and reference fuel blend.

(*k*) *Injection Pump Setting.*—The pump-plunger port should close when the plunger has traveled up 0.075 to 0.090 in. from the base circle of the cam.

NOTE.—This setting is important, as it influences the injection rate. This adjustment is made in the factory and should not require resetting unless it has been tampered with. To check the port closing the following operations shall be made:

(*1*) Remove the discharge valve and spring from the top of the pump, and replace the discharge-valve clamp nut with injection tube arranged conveniently for blowing into.

(*2*) Submerge the end of the fuel drain line in oil.

(*3*) Remove the pump cover, open the fuel pump rack to its wide-open position, and turn the engine until the pump plunger starts up. Then blow on the tube and, at the same time, turn the engine slowly until bubbles disappear at the end of the fuel drain line which is submerged in oil. When bubbles cease, it indicates that the port has just closed. Determine this point carefully by several trials and mark the flywheel.

(*4*) Remove the tube used for blowing into the pump and insert a pin in the discharge-valve hole and resting on the top of the plunger. Arrange a dial indicator resting on the pin to indicate the pump-plunger lift. With the plunger at the bottom of its stroke and the dial gage set on zero, turn the engine until the reference mark, previously placed on the flywheel to indicate the point of port closing, registers. Then read the dial gage, which will indicate the pump-plunger lift from the base circle when the port closes. This lift should be 0.075 to 0.090 in. Any deviation from this value can be adjusted by turning the pump-plunger adjusting screw. After making an adjustment, a new reference mark must be established on the flywheel as outlined in operation (*3*).

(*l*) *Injector Pintle Valve Lift.*—0.004 to 0.006 in.

(*m*) *Injector Cooling-Water Jacket Temperature.*—100 ± 5 F. (38 ± 3 C.).

(*n*) *Exhaust Pipe.*—A separate exhaust pipe used for each engine, made of pipe not less than $1\frac{1}{4}$ in. in inside diameter having a maximum of two ells and a total length not to exceed 20 ft. The use of a short straight-through muffler, or equivalent, with passage not less than the diameter of the exhaust pipe is permissible.

(*o*) *Air-Inlet Temperature.*—Air-inlet temperature maintained at 150 ± 2 F. (66 ± 1 C.).

(*p*) *Clearance Volume.*—The clearance volume measured to the combustion-indicator seat or spot face shall be 72 ± 0.5 ml. with the piston at top dead center, and with the over-travel of the piston past the top of the cylinder adjusted to 0.014 to 0.016 in. by proper cylinder shims. At this volume the micrometer should be set to read 2.000 in. The compression ratio is given by the equation:

$$\text{C.R.} = \frac{18}{L} + 1$$

where L = length of the combustion chamber in inches as measured by the micrometer.

PROCEDURE

Starting and Stopping the Engine

7. While the engine is being turned over by electric motor, the fuel by-pass valve on the injector shall be closed and the compression ratio increased until the engine begins to fire. To stop the engine, the fuel by-pass valve on the injector shall be opened and the electric motor then switched off.

Injection Indicator Contact Setting

8. The injection indicator contact setting shall be made as follows:

(*a*) With the engine not running, adjust injection contact mounting bracket (Fig. 2) until there is about 1/32-in. clearance between the spring leaf and the injector pin.

(*b*) Screw out center adjusting screw.

(*c*) Adjust the lower leaf until it just touches the end of the injector pin. Then apply tension by turning the screw down one more complete turn.

(*d*) Adjust the upper leaf by turning down the upper leaf adjusting screw until the contact points touch. Be sure that the points touch as a result of the leaf-spring tension and not because of force applied downward by the adjusting screw. If the latter should be the case, it is necessary to remove the spring and bend it slightly downward at the outer end. The adjusting screw should then be turned up one complete turn from this position.

(*e*) Adjust the bumper-spring tension in the center adjusting screw by turning down the bumper spring adjusting screw as far as it will go thus locking the two screws so that they move as a unit.

(*f*) With the engine running normally and the neon indicating switch closed, adjust the contact-point gap by turning the bakelite dial clockwise to screw down the center adjusting screw until the neon tube just makes a solid band of light around the flywheel. Then turn back the center adjusting screw 9.4 divisions on the periphery of the bakelite dial (0.002 in.) and lock by means of clamping screws.

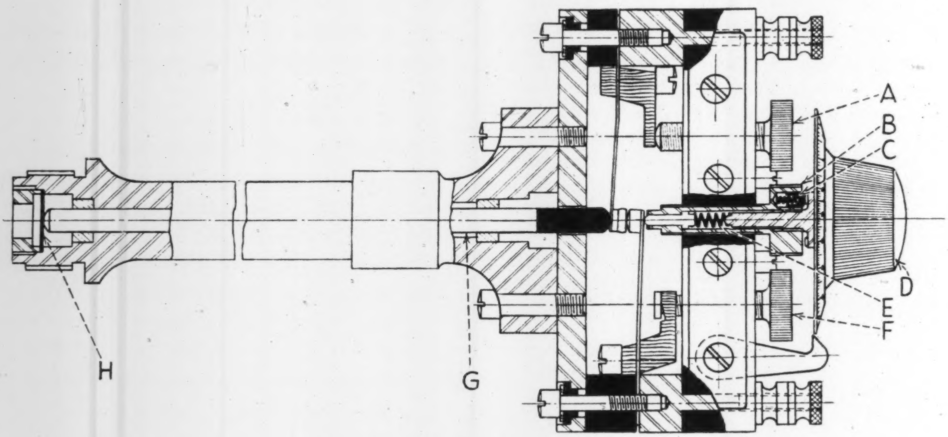

FIG. 1.—Combustion Indicator.

A—Lower Leaf Adjusting Screw.
B—Center Adjusting Screw.
C—Locating Spring.
D—Bumper Spring Adjusting Screw.
E—Bumper Spring.
F—Upper Leaf Adjusting Screw.
G—Pin
H—Diaphragm.

Basic Combustion Indicator Contact Setting

9. With the engine not running, the basic combustion indicator contact setting shall be made as follows:

(*a*) Screw out center adjusting screw (Fig. 1).

(*b*) Adjust the lower leaf until it just touches the end of the combustion indicator pin. Then apply tension by turning the screw down one more complete turn.

(*c*) Adjust the upper leaf by turning the upper leaf adjusting screw down until the contact points just touch. Be sure that the points touch as a result of the leaf-spring tension and not because of force applied downward by the adjusting screw. If the latter should be the case, it is necessary to remove the spring and bend it slightly downward at the outer end. The adjusting screw should then be turned up one complete turn from this position.

(*d*) Adjust the bumper-spring tension in the center adjusting screw by turning down the bumper spring adjusting screw as far as it will go thus locking the two screws so that they move as a unit.

(*e*) Make preliminary contact-point adjustment by screwing down the center adjusting screw until there is a gap of approximately 0.010 in.

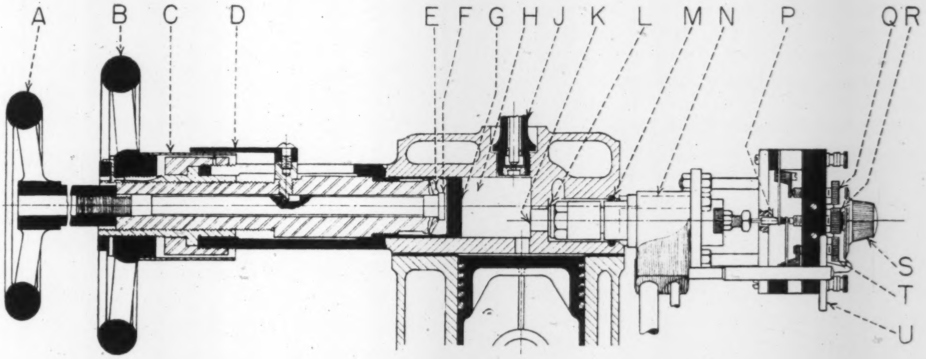

FIG. 2.—Detail of Cylinder Head and Injection Indicator.

A—Locking Wheel.
B—Compression Ratio Adjusting Wheel.
C—Micrometer Scale Sleeve.
D—Micrometer Scale Arm.
E—Locking Washer.
F—Locking Nut.
G—Expansion Plug Head.
H—Combustion Chamber.
J—Combustion Indicator.
K—Injector Tip.
L—Injector Gasket.
M—Rubber Ring.
N—Injector.
P—Injector Pin
Q—Lower Leaf Adjusting Screw.
R—Center Adjusting Screw.
S—Bumper Spring Adjusting Screw
T—Upper Leaf Adjusting Screw.
U—By-Pass Valve Handle

Final Combustion Indicator Contact Setting

10. The final combustion indicator contact setting shall be made with each test fuel as follows:

(*a*) Adjust compression ratio until engine fires normally on sample fuel. This is about two ratios above the lowest operating ratio without misfire.

(*b*) Close neon indicating switch.

(*c*) Check injection angle to make sure it is 13 deg. b.t.d.c. with engine operating on sample fuel. The injection neon tube is displaced 13 deg. on the flywheel to make it flash at t.d.c. when injection actually occurs at 13 deg. b.t.d.c.

(*d*) Make preliminary compression-ratio adjustment until combustion occurs at top dead center as determined by the "hook" method, as follows:

Raise the center adjusting screw from a closed position until the resultant rapid shift in position of the beginning of the flash suddenly changes to a slow rate of shift for a given constant angular motion of the center adjusting screw. This is the "hook" point and the position of the neon flash indicates the start of combustion. Adjust the compression ratio with the handwheel until the combustion flash is brought to the top dead-center mark.

NOTE.—The combustion indicator pin is restrained to follow the diaphragm by means of a leaf spring. The spring pressure is such that, for accelerations up to the maximum rate of pressure rise due to compression pressure, the pin is held on the diaphragm. Thus the gap between the indicator pin and the upper contact is an accurate measure of the deflections of the diaphragm. The position of the diaphragm is a function of the crank angle of the piston and, thus, the position of the upper contact pin determines the angular position of the neon flash.

Near the flat top of the compression-pressure diagram, therefore, a very slight contact-point movement indicates a considerable flywheel angular movement and the neon-tube flash position is very sensitive to the contact-gap adjustment. But when combustion occurs, the very steep pressure rise of the diagram makes the neon flash position very insensitive to contact-gap adjustment. The transition from the sensitive to insensitive gap adjustment range is called the "hook" point.

(*e*) Retard the injection timing to 5 deg. b.t.d.c. for the final-combustion contact-gap adjustment by the "double-flash" method.

(*f*) Adjust the center adjusting screw until two flashes of the combustion-indicator neon bulb on the flywheel are just seen, the first at t.d.c. from compression pressure and the other about 10 deg. a.t.d.c. from combustion pressure. If two flashes are not seen, a lengthening of the combustion flash to a point about 10 deg. a.t.d.c. will be noted, which means the same thing.

(*g*) Turn back the center adjusting screw to raise the upper contact 2.4 divisions (approximately 0.0005 in.) on the bakelite dial where only one flash from combustion will be seen.

Rating

11. (*a*) The injection shall be reset to the rating position of 13 deg. b.t.d.c., which will make the injection flash appear at t.d.c.

(*b*) The final compression ratio shall be adjusted so as to give coincidence of the injection and combustion neon flashes. The combustion contact gap setting shall be checked by the "lingering-flash" method as follows: Screw down the center adjusting screw by 0.0005 in. Cut out injection by opening the injection nozzle by-pass valve, while observing the flashes. The injection flash will go out at once. If the original gap clearance was correct (0.0005 in.), the combustion flash will linger on for one or two revolutions and then die out. If the original gap clearance was too large (more than 0.0005 in.), the combustion flash will go out at once together with the injection flash. If the original gap clearance was too small (less than 0.0005 in.), the combustion flash will stay on indefinitely. If the combustion contact point gap is found to be incorrect, repeat the operations described in Section 10 (*e*), (*f*), (*g*), and in Section 11 (*a*) and (*b*) until the correct adjustment is attained. The combustion chamber length (hand wheel setting) shall then be recorded.

(*c*) From the combustion-chamber length, recorded when the sample fuel has been adjusted to operate at standard conditions with 13-deg. delay period, the cetane number of the unknown sample shall be estimated and two reference-fuel blends selected, differing by not more than eight cetane numbers, which will bracket the unknown sample. When two reference fuels have been found, one of which has a longer delay period and the other a shorter delay period than the sample, alternate readings shall be taken by adjustment of the combustion-chamber length until the neon light flashes coincide at t.d.c.

(*d*) Alternate readings between the sample and the reference fuels shall be taken in the following order:

Sample,
Reference fuel No. 1,
Reference fuel No. 2,
Sample,
Reference fuel No. 2,
Reference fuel No. 1,
Sample.

After changing from one fuel to another, 5 min. shall be allowed before taking a reading to insure complete flushing of the injection system. The average combustion-chamber lengths obtained from the foregoing series shall be used in calculating the cetane number.

Calculation

12. The A.S.T.M. cetane number of the fuel shall be calculated by interpolation from relative combustion-chamber lengths as obtained in Section 11 (*c*). The cetane number shall be reported to the nearest whole number. When the calculated value falls exactly halfway between two whole numbers, the nearest even number shall be reported.

MAINTENANCE

Maintenance

13. The following points are important in maintenance technique.

(*a*) Injector should be inspected and cleaned at least every 8 hr. of operation,

(*b*) Compression plug should be cleaned when it becomes tight due to carbon,

(*c*) Special care should be exercised in keeping the fuels and fuel system clean,

(*d*) A general overhaul, including the grinding of valves, cleaning of piston and rings, and cylinder and cylinder head, should be made every 75 hr. of operation, and

(*e*) Periodic inspection of connecting-rod and main bearings is recommended.

APPENDIX IV

DIESEL-FUEL-OIL CLASSIFICATION

Manufacturers and users of Diesel engines, as well as petroleum refiners and distributors, have long felt the need for some classification differentiating types of fuel oils suitable for Diesel engines. The classification shown in Table I, covering four grades of oil, is offered by Technical Committee E on Diesel Fuel Oils of Committee D-2 on Petroleum Products and Lubricants in an attempt to supply this need. This classification was first presented in June, 1934, and has been revised periodically.

It is not the intention of the committee that this classification should be used as a purchase specification; but rather that fuels purchased be correlated according to the proposed classification in order to determine its usefulness. A series of Diesel-fuel-oil specifications should evolve as a result of this program. Criticism and comment should be forwarded to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 50 West 50th Street, New York, N. Y.

TABLE I.—DIESEL-FUEL-OIL CLASSIFICATION.

Grade of Diesel Fuel Oil	Flash Point, deg. Fahr.	Pour Point, deg. Fahr.	Water and Sediment, per cent by volume	Carbon Residue, per cent by weight	Ash, per cent by weight	Distillation Temperatures, deg. Fahr.		Viscosity at 100 F.			Sulfur, per cent by weight	Corrosion	Alkali and Mineral Acid	Cetane Number
						90 per cent Point	End Point	Kinematic, centistokes		Saybolt Universal, sec.				
	Min.	Max.	Max.	Max.	Max.	Max.	Max.	Min.	Max.	Max.	Max.			Min.
No. 1-D	100 or legal	0	0.05	...	0.01	...	590	...	...	...	0.5	pass	none	45
No. 2-D	110 or legal	20^a	0.05	0.25^b	0.01	675	...	$2.0\ (32.6)^c$	$6.0\ (45.5)^c$	...	1.0	pass	none	42
No. 3-D	110 or legal	35^a	0.1	1.0	0.02	...	...	...	$12\ (65)^c$	...	1.5	...	none	35
No. 4-D	140	35	0.5	3.5	0.05	...	...	...	...	140	2.0	...	none	30
No. 5-D	Oil more viscous than No. 4-D may be specified by referring to No. 5 fuel oil[d] with an agreement between the purchaser and the seller regarding limits for carbon residue and sulfur.													

[a] Lower pour points may be specified whenever required by local temperature conditions to facilitate storage and use.
[b] On 10 per cent residuum.
[c] Equivalent values, Saybolt Universal seconds.
[d] Tentative Specifications for Fuel Oils (A.S.T.M. Designation: D 396) of the American Society for Testing Materials.[1]

METHODS OF TESTING

The properties listed in this classification are determined in accordance with the following methods of test:

(*a*) *Flash Point.*—Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (A.S.T.M. Designation: D 93),[2] except where other methods are prescribed by law.

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[1] 1939 Book of A.S.T.M. Standards, Part. III, p. 586.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 47.

(*b*) *Pour Point.*—Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97).[3]

(*c*) *Water and Sediment.*—Standard Method of Test for Water and Sediment in Petroleum Products by Means of Centrifuge (A.S.T.M. Designation: D 96).[4]

(*d*) *Carbon Residue.*—Standard Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (A.S.T.M. Designation: D 189).[5]

(*e*) *Ash.*—Tentative Method of Test for Ash Content of Petroleum Oils (A.S.T.M. Designation: D 482).[6]

(*f*) *Distillation.*—Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86).[7]

(*g*) *Viscosity.*—Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445),[8] and Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).[9]

(*h*) *Sulfur.*—Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (A.S.T.M. Designation: D 129).[10]

(*i*) *Corrosion.*—Standard Method of Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline (A.S.T.M. Designation: D 130).[11]

(*j*) *Alkali and Mineral Acid.*—Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (A.S.T.M. Designation: D 188).[12]

(*k*) *Cetane Number.*—Proposed Method of Test for Ignition Quality of Diesel Fuels.[13]

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[3] 1939 Book of A.S.T.M. Standards, Part III, p. 102.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 82.

[5] 1939 Book of A.S.T.M. Standards, Part III, p. 98.

[6] *Ibid.*, p. 595.

[7] 1940 Supplement to Book of A.S.T.M. Standards, Parts II and III.

[8] 1939 Book of A.S.T.M. Standards, Part III, p. 647.

[9] 1939 Book of A.S.T.M. Standards, Parts II and III.

[10] 1939 Book of A.S.T.M. Standards, Part III, p. 207.

[11] *Ibid.*, p. 206.

[12] *Ibid.*, p. 617.

[13] See p. 316.

REPORT OF SECTIONAL COMMITTEE
ON
PETROLEUM PRODUCTS AND LUBRICANTS
ASA PROJECT: Z11

Sectional Committee Z11 on Petroleum Products and Lubricants held one meeting during the year: in Atlantic City, N. J., on June 28, 1939.

The Summary of Work in the Field of Petroleum Products which has been in preparation for several years was completed and published in January, 1940, under the title "Evaluation of Petroleum Products." The compilation includes six authoritative papers by leading technologists in the industry, discussing especially important problems on which research work might well be concentrated to develop needed information of service to those concerned with these products. The papers cover: gasoline, lubricating oils, fuel and lubricating oil for spark ignition aircraft engines, petroleum lubricating greases, diesel fuels, and fuel oils.

Two years ago the Study Committee on Boundary Lubrication (J. C. Geniesse, chairman) prepared a set of definitions of terms and concepts used in lubrication; these definitions are still before the sectional committee for comment and criticism.

In line with its function as a reviewing body, the sectional committee has taken action during the year on ten A.S.T.M. methods and one set of definitions for approval either as American standard or American tentative standard, and on the withdrawal of one American tentative standard.

At its meeting held during this annual meeting, on the recommendation of A.S.T.M. Committee D-2, action was taken to refer six methods to the ASA subject to letter ballot of the sectional committee. The recommendation concerning these standards will be submitted to Committee E-10 on Standards for approval.

Respectfully submitted on behalf of the sectional committee,

T. A. BOYD,
Chairman.

R. E. HESS,
Secretary.

REPORT OF COMMITTEE D-3
ON
GASEOUS FUELS

Committee D-3 on Gaseous Fuels held one meeting in 1939, in New York, N. Y., on October 12. In addition, two of its subcommittees met in Detroit, Mich., on March 6, 1940.

While no tentative standards are available as yet, distinct progress has been made in their preparation and a considerable amount of preliminary investigational work completed. This will be evident from the following brief reports on the assignments of the individual subcommittees:

Subcommittee I on Collection of Gaseous Samples (S. S. Tomkins, chairman).—Further steps have been taken in the preparation of individual methods of sampling manufactured, natural, and liquefied petroleum gases. Attention has also been devoted to the formulation of definitions of terms in common use.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman).—Further work has been conducted at the National Bureau of Standards on the testing of small wet gas meters under conditions similar to those of operation, including the preparation of necessary equipment. Tests were completed to furnish a basis for the calculation of the correction for liquid film adhering to a volumetric standard, such as a meter prover bell or a cubic foot standard.

A paper was published during the year giving results of work conducted on saturation of gases by laboratory wet test meters.[1]

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. B. Harper, chairman).—Experiments have been conducted on applications of the "humidity control" method in calorific value determinations and it has been found generally satisfactory, the results agreeing closely with those obtained by use of the "humidity correction" method. A critical study has also been made of "humidity correction" methods and recommended procedures. It is planned later to embody both methods in a proposed standard method for routine determinations of the calorific value of gaseous fuels.

A memorandum covering specifications for mercurial thermometers for gas flow calorimetry was prepared and submitted to the members for criticism before supplying it to manufacturers of such thermometers.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—Tests have been continued, by the National Bureau of Standards as planned, on eleven instruments in common use for specific gravity determination, submitted by the manufacturers for this purpose. A synthetic typical manufactured gas was prepared and its specific gravity determined. A series of ten determinations conducted on each of the eleven instruments has provided data for the computation of reproducibility and accuracy under each of several conditions of temperature and humidity. All instruments have been tested at a normal condition of 77 F. and 35 per cent relative humidity. The chart instruments have been tested at two other temperatures to determine the effect of temperature at constant relative humidity. Instruments using the surrounding air as a standard of reference have been tested under two additional humidity conditions to determine the effect of the water content of the air at constant temperature. Further tests are in progress on all instruments at varying temperatures with the humidity constant.

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[1] F. A. Smith and J. H. Eiseman, "Saturation of Gases by Laboratory Wet Test Meters," *Journal of Research*, Nat. Bureau Standards, Vol. 23, No. 3, September, 1939, p. 345.

Subcommittee V on Determination of Impurities of Gaseous Fuels (E. F. Pohlman, chairman).—Tentative methods for the determination of total sulfur in gaseous fuels have been studied and are receiving principal consideration. A review has been completed of the present methods for the determination of tar fog and condensible oils and vapors, gums, cyanogen, and ammonia. Tentative methods for the determination of these impurities in gaseous fuels are now in preparation.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (A. W. Gauger, chairman).—Two methods for water vapor determination are under development, one primarily for field work and the other for a laboratory standard. The first employs a change in color of cobalt salts in organic solvents on the addition of water. A portable instrument has been constructed employing two vacuum photocells in a bridge circuit which has been found capable of giving consistent readings under the test conditions selected. The laboratory method employs absorption of a wave length in the near infrared by water vapor A spectrometer. has been constructed and is now operating very satisfactorily in the visible region by use of a photocell as the radiation receiver. Further tests are awaiting the completion of additional equipment required.

A progress report on the experimental work conducted was presented at the Joint Production and Chemical Conference of the American Gas Association in New York, N. Y., on May 20, 1940.

Subcommittee VII on Complete Analysis or Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman).—The first standard samples of fuel gas of the carburetted water-gas type were prepared. Cylinders were purchased and fitted up for use as containers for shipment of this gas to various laboratories. Following previously made arrangements, samples of such gas have been sent out to approximately 35 laboratories together with specific instructions covering the analysis to be made. A second standard sample consisting of typical natural gas will be sent to these cooperating laboratories on return of the sampling containers used in the first sample.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, A. W. Gauger
Vice-Chairman, A. C. Fieldner
Secretary, R. M. Conner

This report has been submitted to letter ballot of the committee which consists of 32 members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

R. M. CONNER,
Secretary.

REPORT OF COMMITTEE D-4

ON

ROAD AND PAVING MATERIALS

Committee D-4 on Road and Paving Materials has held two meetings during the past year: in Atlantic City, N. J., on June 28, 1939, and in Detroit, Mich., on March 7, 1940.

Subsequent to the 1939 annual meeting Committee D-4 presented to the Society, through Committee E-10 on Standards, the following recommendations:

New Tentative Standards:

Specifications for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544 – 39 T),

Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 – 39 T), and

Method of Test for Sieve Analysis of Mineral Filler (D 546 – 39 T).

Revision of Tentative:

Specifications for Asphalt Filler for Brick Pavements (D 241 – 39 T), and

Method of Test for Loss on Heating of Oil and Asphaltic Compounds (D 6 – 39 T).

These recommendations were accepted[1] by Committee E-10 on August 24, 1939, and the new and revised tentative standards appear in the 1939 Book of A.S.T.M. Standards.[2]

In connection with the Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (D 6 – 39 T), Committee D-4 feels that the results of comparative tests made by Subcommittee B-18 on Loss on Heating, then under the chairmanship of B. A. Anderton, should be made available to all who have an interest in this test. A brief explanation of the subcommittee's work together with a tabulation of test results is therefore appended to this report.

At the March meeting of Committee D-4, the following officers were elected for the ensuing term of two years, beginning at the close of the June, 1940, meeting of the committee:

Chairman: J. E. Myers,
First Vice-Chairman: H. H. Scofield,
Second Vice-Chairman: A. T. Goldbeck,
Third Vice-Chairman: E. O. Rhodes, and
Secretary: Prévost Hubbard.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee B-15 on Sampling Road Materials (W. H. Mills, chairman).—This subcommittee has thoroughly revised the Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 – 39 T), which were last year withdrawn from the status of standard for the purpose of revision, and the revised method will be submitted to letter ballot of Committee D-4 for presentation to the Society later in the year through Committee E-10 on Standards.[3]

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-4 reported the following results of the letter ballot vote from a committee membership of 122: D 544 – 39 T, affirmative 78, negative 0, ballots marked "not voting" 13; D 545 – 39 T, affirmative 59, negative 1, ballots marked "not voting" 32; D 546 – 39 T, affirmative 60, negative 1, ballots marked "not voting" 31; D 241 – 39 T, affirmative 65, negative 4, ballots marked "not voting" 23; D 6 – 39 T, affirmative 73, negative 1, ballots marked "not voting" 17.

[2] 1939 Book of A.S.T.M. Standards, Part II, pp. 1015, 1017, 1021, 1033, 1045.

[3] See Editorial Note, p. 332.

Subcommittee B-17 on Emulsion Tests (J. S. Miller, chairman.—During the past year this subcommittee has conducted an exhaustive investigation which has resulted in the development of the following three new tests which are receiving further study before being presented to the Society as proposed Tentative Methods of Test for Modified Miscibility of Emulsified Asphalts, Dehydration Test of Emulsified Asphalts, and Test for Portland Cement Mixing of Emulsified Asphalts.

Subcommittee C-3 on Specifications for Liquid Asphaltic Road Materials (W. J. Emmons, chairman).—For some time past this subcommittee has been actively engaged in the development of specifications for cut-back asphalts and has recommended to Committee D-4, new tentative specifications covering six grades of rapid curing cut-back asphalts and six grades of medium curing cut-back asphalts which will be submitted to letter ballot for presentation to the Society later in the year through Committee E-10.[3]

Subcommittee C-4 on Specifications for Emulsified Asphalts (J. E. Myers, chairman).—This subcommittee has recently developed new proposed tentative specifications for a slow setting emulsified asphalt for fine aggregate mixes which will receive further study before presentation to the Society.

Subcommittee D-2 on Highway Traffic Markers (H. F. Clemmer, chairman).—During the past year this subcommittee has developed new proposed tentative specifications for traffic paint which are being reviewed by Committee D-1 on Paint, Varnish, Lacquer, and Related Products before definite action by Committee D-4.

Subcommittee D-3 on Expansion Joint Materials (H. M. Milburn, chairman).—This subcommittee has developed proposed Specifications and Methods of Test for Preformed Expansion Joint Fillers for Concrete (Bituminous Type) which it is felt should be published as information with a view to securing comments and criticisms. These proposed specifications and methods of test are therefore appended hereto.[4]

I. NEW TENTATIVE STANDARDS

Tentative Specifications for Materials for Stabilized Base Course, and for Materials for Stabilized Surface Course.—Methods of stabilizing soils and soil aggregate mixtures are engaging the attention of highway engineers throughout the country to a very great extent, and there is definite need for standardization of materials which are to be used for this purpose. Committee D-4, through its Subcommittee D-5 on Soils and Soil Mixtures for Highway Construction (C. A. Hogentogler, chairman), has devoted a considerable amount of study to this subject in relation to results obtained by numerous state highway organizations, and the specifications for materials for stabilized base course, and for stabilized surface course are the outcome of this study. The committee recommends that these specifications be accepted for publication as tentative.[5]

II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following two tentative specifications[6] be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for:

Quick-Setting Emulsified Asphalt (for Penetration and Surface Treatment) (D 401 - 36 T), and

Asphalt Plank (D 517 - 38 T).

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[4] See pp. 335, 336.

[5] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 272 and 274, respectively.

[6] 1939 Book of A.S.T.M. Standards, Part II, pp. 1031, 1035.

III. WITHDRAWAL OF TENTATIVE STANDARD

The Deval test for abrasion of gravel has been largely supplanted by the Los Angeles abrasion test (C 131 – 39) which, on the recommendation of Committee D-4 and Committee C-9 on Concrete, was adopted as standard last year. It is therefore recommended that the Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine (D 289 – 37 T)[7] be withdrawn.

IV. CHANGES IN STANDARDS

Committee D-4 recommends that the following addition be incorporated in the Standard Methods of Testing Emulsified Asphalts (D 244 – 39)[8] and accepted as an editorial correction as the change does not in any way alter the intent or meaning of the test methods but is in the interest of simplification:

Section 5 (a).—In the note insert after the first sentence, a new sentence to read as follows:

> Either the still shown in Fig. 1 or that shown in Fig. 2 may be used interchangeably.

At its meeting on June 27, 1940, the committee approved, jointly with Committee D-8 on Bituminous Waterproofing and Roofing Materials, the following revision in the Standard Methods of Testing Emulsified Asphalts (D 244 – 39).[8] This change in the calibration of the centrifuge tube was made on the recommendation of the Section on Volumetric Glassware, of Technical Committee XII, of Committee E-1 on Methods of Testing:

Section 8 (f).—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> The trap shall be made of well-annealed glass constructed in accordance with Fig. 3 (*c*) and shall be graduated from 0 to [25 ml.] *2 ml.* in 0.1-ml. divisions, *and from 2 to 25 ml. in 0.2-ml. divisions.*

The committee also approved at its June, 1940, meeting, jointly with Committee D-2 on Petroleum Products and Lubricants, the following revision in the Standard Methods of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 – 30).[8] This revision in the calibration of the centrifuge tube used in the test is being made to bring the method into conformity with the change in Standard Methods D 244 – 39 given above.

Section 5 (a).—In the note following this section change the last sentence to read as follows, by the addition of the italicized words and figures and the omission of those in brackets:

> This tube shall be graduated from 0 to [25 ml.] *2 ml.* in 0.1-ml. divisions *and from 2 to 25 ml. in 0.2-ml. divisions* (see Fig. 3 (*c*) of the Standard Methods of Testing Emulsified Asphalts (A.S.T.M. Designation: D 244) of the American Society for Testing Materials).

V. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

Committee D-4 recommends that the following specifications be continued as tentative for the reasons given below:

Tentative Specifications for Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 37 T).—It appears likely that it will be desirable to revise the size requirements which were made to conform with the Simplified Practice Recommendation for Coarse Aggregates (R 163 – 36), approved by the Division of Simplified Practice in 1936. The Simplified Practice Recommendations were, however, revised on May 25, 1939, and certain questions that have since arisen indicate the possibility of further revisions. In view of this possibility, and the fact that a revision of Specifications D 448 – 37 T will also require revision of a number of other A.S.T.M. specifications that make use of standard sizes of coarse aggregate, it is thought inadvisable to recommend that the present tentative specifications be adopted as standard.

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[7] 1939 Book of A.S.T.M. Standards, Part II, p. 1023.

[8] *Ibid.*, p. 400.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Materials for Stabilized Base Course...........	66	4	19
Spec. for Materials for Stabilized Surface Course........	65	5	19
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Quick-Setting Emulsified Asphalt (for Penetration and Surface Treatment) (D 401 - 36 T)[a].	55	7	27
Spec. for Asphalt Plank (D 517 - 38 T)...............	56	0	33
III. WITHDRAWAL OF TENTATIVE STANDARD			
Test for Abrasion of Gravel by Use of the Deval Machine (D 289 - 37 T)[b]....	60	9	20

[a] The classified vote on the adoption as standard of the Tentative Specifications for Quick-Setting Emulsified Asphalts (for Penetration and Surface Treatment) (D 401 - 36 T) was as follows: Affirmative: 19 producers, 27 consumers, 9 general interests; negative: 6 producers, 1 consumer, 0 general interests.

[b] The classified vote on the withdrawal of the Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine (D 289 - 37 T) was as follows: Affirmative: 20 producers, 28 consumers, and 12 general interests; negative: 2 producers, 7 consumers, and 0 general interests.

Tentative Specifications for Crushed Slag and Crushed Stone.—The Tentative Specifications for Crushed Slag for Water-Bound Base and Surface Courses (D 488 - 38 T), for Crushed Stone for Water-Bound Base and Surface Courses (D 489 - 38 T), and for Crushed Slag for Bituminous Concrete Base and Surface Courses (D 485 - 38 T) are being continued as tentative for the reasons given above for Specifications D 448 - 37 T. Moreover, a revision of the physical property requirements for crushed stone for water-bound base and surface courses is at present being considered by Committee D-4 and will probably be referred to letter ballot of the committee for presentation to the Society in the near future.

Tentative Specifications for Tar (D 490 - 38 T).—Committee D-4 is attempting to coordinate these specifications with similar specifications for tar adopted by the American Association of State Highway Officials.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 121 members; 89 members returned their ballots, with the results shown in Table I.

This report has been submitted to letter ballot of the committee which consists of 121 members; 89 members returned their ballots, of whom 82 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. F. KELLEY,
Chairman.

PRÉVOST HUBBARD,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-4 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:
- Cut-Back Asphalt (Rapid Curing Type) (D 597 - 40 T),
- Cut-Back Asphalt (Medium Curing Type) (D 598 - 40 T),

Revision of:
- Tentative Specifications for Crushed Stone for Water-Bound Base and Surface Courses (D 489 - 38 T), and
- Tentative Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials, Including Some Material Survey Methods (D 75 - 39 T).

These recommendations were accepted by Committee E-10 on August 28, 1940, and the new and revised tentative standards appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 283, 281, 270, and 276.

APPENDIX I

REPORT ON COMPARATIVE TESTS OF LOSS ON HEATING

In 1939 Committee D-4 on Road and Paving Materials submitted to the Society, for acceptance by Committee E-10 on Standards, a revised Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (D 6 – 39 T).[1] The most important revisions were in Section 2, in which requirements for the oven are included. Requirements in the former Standard Method D 6 – 33[2] were based on an oven which is no longer manufactured, and were so stated as to exclude many ovens now in use and entirely satisfactory for the purpose. Criticism of these requirements has been received by the committee for several years from numerous testing engineers and manufacturers of ovens. The proposed revision was designed to permit the use of modern ovens having inner and outer doors and provided with improved means for ventilation.

In connection with requirements for ventilating openings, Subcommittee B-18 on Loss on Heating reviewed tests made by a sectional committee of the Committee on Materials, of the American Association of State Highway Officials, which showed that the amount of air passing through the oven during the loss on heating test is of little importance within practical limits. A series of comparative tests was also conducted for the subcommittee at the laboratory of the New Jersey State Highway Commission, using three ovens of different size and design, all of which conformed to the revised requirements published in 1939.[1] Samples were tested in duplicate at the same time in each of the three ovens, recording the size and arrangement of openings provided for ventilation. The tests showed excellent agreement with samples of widely different character. Results of these tests pertinent to the A.S.T.M. method are summarized in Table I. It may be concluded that the degree of ventilation in ovens through which the heated air is circulated by natural convection is not a critical feature of the test method within practical limits.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 1045.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 1071.

TABLE I.—RESULTS OF COMPARATIVE TESTS OF LOSS ON HEATING MADE BY LABORATORY OF STATE HIGHWAY COMMISSION, STATE OF NEW JERSEY.

Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6 – 39 T),[1] 50 g., 163 C. (325 F.), 5 hr.

		Oven Serial Number	300-102			320-2			2708		
		Dimensions of Heating Chamber:									
		Height in.	13			18			11¾		
		Width in.	13⅛			18 5/16			11¾		
		Depth in.	14⅛			18⅛			12¾		
		Openings for Ventilation:									
		Bottom, outside	156 3/16 in. Holes, Spaced 1 in. Apart			One ¾ in. Hole[a]			One ¾ in. Hole[a]		
		Bottom of heating chamber	132 ¼ in. Holes, Spaced 15/16 in. Apart						One ¾ in. Hole in each of 2 Sides		
		Top of heating chamber	144 ⅛ in. Holes, Spaced 1 in. Apart			One ¾ in. Hole in each of 2 Sides			One ¾ in. Hole in each of 2 Sides[b]		
Serial Number	Material	Tests	1	2	Average	1	2	Average	1	2	Average
205747	Asphaltic Oil Grade C.C.	Loss on Heating, per cent	49.9	49.9	49.9	49.6	49.7	49.6	50.1	49.9	50.0
		Penetration of Residue[c]			21			21			21
213123	Asphaltic Oil Grade L.K.	Loss on Heating, per cent	34.0	34.1	34.0	34.0	33.9	33.9	33.9	33.9	33.9
		Penetration of Residue[c]			57			58			58
208226	Asphaltic Oil Grade MC-1	Loss on Heating, per cent	24.7			24.9			24.8		
		Float Test of Residue[d] at 50 C., sec.	43			44			44		
212828	Asphaltic Oil Grade H.K.	Loss on Heating, per cent	23.3	23.2	23.3	23.3	23.3	23.3	23.5	23.5	23.5
		Penetration of Residue[c]			73			73			73
213177	Asphaltic Oil Grade H.K.	Loss on Heating, per cent	24.8	24.8	24.8	24.7			24.8		
		Penetration of Residue[c]			74	74			74		
213124	Asphaltic Oil Grade H.K.	Loss on Heating, per cent	23.2	23.1	23.2	23.2	23.2	23.2	23.2	23.2	23.2
		Penetration of Residue[c]			76			76			76
212686	Asphaltic Oil Grade S.C.O.	Loss on Heating, per cent				14.9			14.3		
		Float Test of Residue[d] at 50 C., sec.				18			18		
212827	Asphaltic Oil Grade S.C.O.	Loss on Heating, per cent	16.5	16.3	16.4	16.7	16.7	16.7	16.5	16.9	16.7
		Float Test of Residue[d] at 50 C., sec.			21			21			21
213687	Asphalt Cement Bermudez	Loss on Heating, per cent	1.00	1.01	1.01	1.06	1.03	1.05	0.99	0.97	0.98
	85 to 100 Penetration	Penetration of Residue[c]			61			61			62
213681	Asphalt Cement Trinidad	Loss on Heating, per cent	0.55	0.56	0.56	0.55	0.57	0.56			
	85 to 100 Penetration	Penetration of Residue[c]			79			79			
213617	Asphalt Cement Hy. Mexican Crude	Loss on Heating, per cent	0.047	0.046	0.047	0.043	0.047	0.045	0.045	0.046	0.046
	85 to 100 Penetration	Penetration of Residue[c]			73			73			74
213684	Asphalt Cement Columbian Crude	Loss on Heating, per cent	0.008	0.006	0.007	0.007	0.006	0.006	0.006	0.006	0.006
	85 to 100 Penetration	Penetration of Residue[c]			70			69			69
213688	Waterproofing Asphalt Cement	Loss on Heating, per cent	0.054	0.050	0.052	0.061	0.053	0.057	0.054	0.050	0.052
		Penetration of Residue[c]			27			27			27

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[a] Closed during tests.

[b] Only one hole open during tests.

[c] Tests for penetration of residue were made at 25 C. (77 F.) under a load of 100 g. for 5 sec. in accordance with the Standard Method of Test for Penetration of Bituminous Materials (D 5 – 25), 1939 Book of A.S.T.M. Standards, Part II, p. 412.

[d] Standard Method of Float Test for Bituminous Materials (D 139 – 27), 1939 Book of A.S.T.M. Standards, Part II, p. 409.

APPENDIX II

PROPOSED SPECIFICATIONS FOR PREFORMED EXPANSION JOINT FILLER FOR CONCRETE[1]

BITUMINOUS TYPE

These are proposed specifications and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These specifications cover bituminous preformed expansion joint filler for use in concrete construction.

Manufacture

2. Bituminous preformed joint filler shall be of asphaltic or tar composition of approved quality. The bitumen shall be uniformly impregnated with suitable filler, to reduce to a minimum its brittleness at low temperatures.

Character of Strips

3. The preformed strips of expansion joint filler shall be of such character as not to be deformed or broken by twisting, bending, or other ordinary handling when exposed to atmospheric conditions. Pieces of the joint filler which have been damaged shall be rejected. Thin strips of stiffener will be allowed.

Physical Properties

4. The joint filler shall conform to the following requirements as to physical properties:

Absorption, max., per cent by weight	5
Distortion, max., in.	1
Brittleness	the joint filler shall not crack or shatter when subjected to the brittleness test

Dimensions and Permissible Variations

5. The preformed strips shall conform to the dimensions specified or shown on the plans. Strips of the joint filler which do not conform to the specified dimensions, within the permissible variations of plus $\frac{1}{16}$ in. in thickness, plus or minus $\frac{1}{8}$ in. in depth, and plus or minus $\frac{1}{4}$ in. in length, shall be rejected.

Sampling

6. (*a*) *Size of Samples.*—Each sample shall consist of sufficient material to provide at least three test specimens measuring 2 by 6 in.

(*b*) *Number of Samples.*—At least one representative sample shall be selected from each shipment of 1000 sq. ft. or fraction thereof of each thickness ordered.

(*c*) Samples shall be packed for transportation in such a manner that there will be no danger of distortion or breakage.

Methods of Testing

7. The properties enumerated in these specifications shall be determined in accordance with the Proposed Methods of Testing Preformed Expansion Joint Filler for Concrete (Bituminous Type) of the American Society for Testing Materials.[2]

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[1] These proposed specifications are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

[2] See p. 336.

APPENDIX III

PROPOSED METHODS OF TESTING PREFORMED EXPANSION JOINT FILLER FOR CONCRETE[1]

BITUMINOUS TYPE

These are proposed methods and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These methods cover the following tests for preformed, bituminous type, expansion joint filler for concrete: absorption, brittleness, and distortion.

Absorption

2. (*a*) A test specimen measuring 2 by 6 in. shall be cut from the expansion joint filler in such a manner that all edges are freshly cut.

(*b*) The specimen shall be weighed, immersed in water for 24 hr., removed, and the surface water wiped off with a slightly dampened cloth. The specimen shall then be quickly weighed, and the percentage of absorption calculated.

Brittleness

3. (*a*) A test specimen measuring 2 by 6 in. shall be cut from the expansion joint filler parallel to the lay of the fiber.

(*b*) Prior to testing, the specimen shall be maintained at a temperature of 4 to 6 C. in water for at least 2 hr. The specimen shall then be clamped between two boards in such a manner that it cantilevers 3½ in. The specimen between the boards shall be supported horizontally and may be held in any suitable manner. A cast iron ball, weighing 0.95 lb. and having a diameter of 1⅞ in., shall be suspended above the specimen by a cord tied to an eyelet soldered to the ball. The ball shall be suspended above the center of the projecting portion of the specimen at a height in accordance with the following requirements:

Thickness of Specimen, in.	Height of Fall of Ball, in.
¼	8
⅜	12
½	16
¾	24
1	32

The ball shall be released by burning the string above the eyelet.

Distortion

4. (*a*) A test specimen measuring 2 by 6 in., absolutely flat and straight, shall be cut parallel to the lay of the fiber.

(*b*) The specimen shall be clamped between two blocks in such a manner that it cantilevers 3⅓ in. The clamp with the specimen shall then be placed in an oven maintained at 125 F. for 2 hr. At the end of the 2-hr. period the deflection of the specimen from the horizontal shall immediately be measured.

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[1] These proposed methods are under the jurisdiction of the A.S.T.M. Committee D-4 on Road and Paving Materials.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE

Committee D-5 on Coal and Coke held a meeting in Detroit, Mich., on March 5, 1940; another meeting will be held at the annual meeting of the Society in June, 1940, in Atlantic City, N. J.

Subsequent to the 1939 annual meeting, Committee D-5 presented to the Society through Committee E-10 on Standards a proposed Tentative Method of Test for Index of Dustiness of Coal and Coke (D 547 - 39 T). The new method was accepted[1] for publication as tentative on August 24, 1939, and appears in the 1939 Book of A.S.T.M. Standards.[2]

During the year the committee lost by death R. S. MacPherran who served as a member for many years. The present membership of the committee is 44, of whom 12 are classified as producers, 13 as consumers, and 19 as general interest members.

I. Revision of Tentative Standard

The committee recommends that the Tentative Method of Sampling Coals Classed According to Ash Content (D 492 - 38 T)[3] be revised and continued as tentative. As these revisions are quite extensive the method is presented in its revised form.[4]

II. Adoption as Standard of Tentative Revisions of Standards

The committee recommends that the tentative revisions published in 1937[5] of the following two methods be approved for reference to letter ballot of the Society for adoption as standard:

Standard Methods of:[6]

Sampling Coal for Analysis (D 21 - 16), and

Laboratory Sampling and Analysis of Coal and Coke (D 271 - 37), Sections 2 (*e*), (*g*), (*p*), 3 (*a*), and 5 (*c*) only; the tentative revision of Sections 27 to 31, and of Section 43 is being continued as tentative.

These recommendations have been submitted to letter ballot of the committee which consists of 44 members; 37 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. Revision of Tentative Standard			
Sampling Coals Classed According to Ash Content (D 492 - 38 T)	36	0	1
II. Adoption as Standard of Tentative Revisions of Standards			
Sampling Coal for Analysis (D 21 - 16)	36	0	1
Laboratory Sampling and Analysis of Coal and Coke (D 271 - 37)	36	0	1

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee D-5 reported results of the letter ballot vote as follows: Of a total membership of 44, 40 members returned their ballots, of whom 34 voted affirmatively, 0 negatively, and 6 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part III, p. 580.

[3] *Ibid.*, p. 559.

[4] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 191.

[5] 1939 Book of A.S.T.M. Standards, Part III, p. 1135.

[6] 1939 Book of A.S.T.M. Standards, Part III, pp. 11, 15.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (W. A. Selvig, chairman).—A meeting of the subcommittee was held on March 4, 1940, in Detroit, Mich. Data have been collected regarding the composition of the atmosphere surrounding the ash cones in the standard method for determining the softening temperature of coal ash when operating the gas furnace with various gases. The revision published as tentative in 1938[7] of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 37) specifies the nature of this atmosphere. Since trouble is being experienced with some city gases in maintaining the specified limits of reducing to oxidizing gases, this revision is being continued as tentative pending further consideration by the subcommittee.

Experimental work has been conducted on methods suitable for the determination of ash content of coals containing pyrite and calcite. Difficulty in obtaining check determinations of ash is often experienced with such coals because of variable amounts of sulfur fixed in the ash as calcium sulfate, dependent on the manner in which the coal sample is ignited for the ash determination. The subcommittee proposes to recommend a suitable procedure for ashing such coals.

Subcommittee II on Nomenclature and Definitions (H. C. Porter, chairman).—At a meeting of Committee D-5 held in Detroit, Mich., on March 5, 1940, this subcommittee recommended for adoption as standard a revision of the Tentative Definition of the Term Coke (D 121 – 26 T), as follows:

Change the definition of coke from its present form: namely,

> *Coke.*—The infusible, cellular, coherent solid material obtained from coal as the residue from its destructive distillation.
>
> NOTE 1.—Such material obtained from the destructive distillation of petroleum or petroleum residues is known as "petroleum coke."
>
> NOTE 2.—Such material obtained from the destructive distillation of coal tar pitch is known as "pitch coke."

to read as follows:

> *Coke.*—(*a*) The solid, cellular, coherent residue from destructive distillation of bituminous coal, said residue having such physical structure as results from the hardening of a fused or semiliquid mass.
>
> (*b*) If used with appropriate qualifying adjective, a similar residue from petroleum, pitch, and some other carbonaceous materials.

This recommendation was submitted to letter ballot of Committee D-5 and approved by a vote of 32 affirmative, 2 negative, and 3 ballots marked "not voting." However, in deference to a written request dated June 24, 1940, from the Executive Committee of the Society addressed to the chairman of Committee D-5 it was voted at a meeting of Committee D-5 held on June 27, 1940, not to present this revised definition at the annual meeting of the Society.

The Tentative Definitions of the Terms Gross Calorific Value and Net Calorific Value of Fuels (D 407 – 35 T) are being continued as tentative pending agreement whether "calorific value" or "heat of combustion" is the most suitable term.

Subcommittee VII on Pulverizing Characteristics of Coal (John Van Brunt, chairman).—The Tentative Methods of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 37 T) and by the Ball-Mill Method (D 408 – 37 T) are to be continued as tentative as the subcommittee has not reached agreement as to whether one or both of these methods should be adopted as standard.

Subcommittee VIII on Foundry Coke Specifications (J. T. MacKenzie, chairman).—This subcommittee held a meeting in Detroit, Mich., on March 4, 1940. The subcommittee is not ready at this time to recommend any revisions of the Standard Specifications for Foundry Coke (D 17 – 16). Consideration is being given to the properties of coke which should be included in specifications and how these properties should be measured and evaluated.

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[7] 1939 Book of A.S.T.M. Standards, Part III, p. 1136.

Subcommittee XI on Coal Friability (R. E. Gilmore, chairman).—Since the subcommittee is considering revisions of the Tentative Methods of Drop Shatter Test for Coal (D 440 – 37 T) and Tumbler Test for Coal (D 441 – 37 T) these tests are being continued as tentative.

Subcommittee XIII on Coal Sampling (H. F. Hebley, chairman).—Two meetings were held during the past year, one in Columbus, Ohio, on October 4, 1939, and the other in Detroit, Mich., on March 5, 1940. The Tentative Method of Sampling Coals Classed According to Ash Content (D 492 – 38 T) is being revised and continued as tentative, as mentioned earlier in this report. The ultimate objective of the subcommittee is to combine this method with the Standard Method of Sampling Coal for Analysis (D 21 – 16). The subcommittee is recommending for adoption as standard the tentative revision of the Standard Method of Sampling Coal for Analysis (D 21 – 16) and that part of the tentative revision of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 37) which pertains to sampling procedures. These revisions are necessary so that the standards will agree with the method of crushing and reducing coal samples prescribed in the Tentative Method of Sampling Coals Classed According to Ash Content (D 492 – 38 T).

Subcommittee XIV on Dustiness of Coal and Coke (O. O. Malleis, chairman).—A few criticisms of a minor nature have been received since the publication of the Tentative Method of Test for Index of Dustiness of Coal and Coke (D 547 – 39 T). The subcommittee recommends that the method be continued as tentative to afford further opportunity for trial.

Subcommittee XV on Plasticity and Swelling of Coal (C. C. Russell, chairman).—This subcommittee held a meeting on June 26, 1939, in Atlantic City, N. J. Cooperative tests have been made by various laboratories using the crucible swelling test issued by British Standards Institution. The test appears to give information of value regarding caking characteristics of coals in fuel beds. No further experimental work, since that reported last year, has been conducted by the subcommittee on methods for the determination of expansion properties of coals during coking. As various authorities are not in agreement on methods of testing coal expansion, it is not advisable at this time to attempt standardization of such test methods. The subcommittee is keeping in close touch with all experimental work being done on the testing of coal expansion during carbonization. During the year a paper was prepared on the measurement of pressures developed during coal carbonization.[8]

Subcommittee XVI on Ignitability of Coal and Coke (R. A. Sherman, chairman).—A meeting of the subcommittee was held on June 27, 1939, in Atlantic City, N. J., and on March 4, 1940, in Detroit, Mich. Cooperative tests with various laboratories have been made using a method for the determination of ignition temperature of coal and coke developed in the Coal Research Laboratory of the Carnegie Institute of Technology.[9] The cooperating laboratories were the Battelle Memorial Institute, the Coal Research Laboratory of Carnegie Institute of Technology, and the Fuel Research Laboratories of the Canadian Bureau of Mines. The subcommittee plans to prepare the method for publication in the form of a proposed tentative standard.

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[8] C. C. Russell, "Measurement of Pressures Developed During the Carbonization of Coal," *Technical Paper 1118*, Am. Inst. Mining and Metallurgical Engrs. (1939).

[9] J. J. S. Sebastian and M. A. Mayers, "Coke Reactivity—Determination by a Modified Ignition Point Method," *Contribution 59*, Coal Research Laboratory, Carnegie Inst. Technology, October, 1937.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot[10] of the committee which consists of 44 members; 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

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[10] See Editorial Note below.

EDITORIAL NOTE

The report of Committee D-5 as written for presentation to the Society, approved by letter ballot of the committee, and preprinted contained a recommendation that the proposed revision of the definition of the term "Coke" printed on page 338 be adopted as standard by the Society. However, in deference to a suggestion from the Executive Committee of the Society that this definition be not adopted, Committee D-5 voted at its meeting on June 27, 1940, not to present the revised definition. The recommendation was withdrawn from the report in presenting the report to the Society, and the portion of the report under the heading "Subcommittee II on Nomenclature and Definitions" page 338 was revised to the form in which it is now published. The letter ballot vote of the committee on its report, recorded above, was taken on the report as preprinted and not as presented and subsequently published herein.

MINORITY REPORT ON ANNUAL REPORT OF COMMITTEE D-5

I should like to be placed on record as objecting to and voting against both the presentation and the acceptance of the 1940 annual report of Committee D-5 in the form as amended by the committee on June 27, 1940, without letter ballot vote.

While I am in accord with the opinion of the majority of the committee that we should accede to the expressed wish of the Executive Committee that the proposed definition of the term "coke" in the annual report "be not adopted," since members representing the petroleum industry have said this definition would cause injury to that industry, yet I am strongly of the opinion that the committee should have followed a more regular procedure, and one correctly informative of its previous actions and those of Committee E-8 on Nomenclature and Definitions recommending this definition. To this end, Committee D-5 should have presented the original report as approved by letter ballot and preprinted, and then have asked, at the Society's meeting, to withdraw the recommended standard definition in deference to the Executive Committee's suggestion.

Respectfully submitted,

HORACE C. PORTER

REPORT OF COMMITTEE D-6

ON

PAPER AND PAPER PRODUCTS

Committee D-6 on Paper and Paper Products held one meeting during the year: on June 27, 1939, in Atlantic City, N. J., during the annual meeting of the Society.

Cooperative relations have been continued between Committee D-6 and Committee D-9 on Electrical Insulating Materials in regard to the interest of the latter committee in electrical insulating paper and methods of testing paper which are being developed by Committee D-6. In this connection Committee D-9 is represented on Committee D-6 by R. W. Chadbourne, and Committee D-6 is represented on Committee D-9 by L. S. Reid.

Cooperation between the Society and the Technical Association of the Pulp and Paper Industry has been successfully carried out during the year through the Joint A.S.T.M.-T.A.P.P.I. Committee on Paper Testing Methods.

By action of the Advisory Committee, the organization of Subcommittee III on Paper Bags (R. M. Bates, chairman) has been discontinued; Mr. Bates has been appointed a member of Subcommittee II on Significance of Test Methods.

During the year 3 new members were elected, and 2 resignations were accepted, leaving a total membership of 47, of whom 18 are classified as producers, 15 as consumers, and 14 as general interest members.

Subsequent to the 1939 annual meeting, Committee D-6 presented to the Society through Committee E-10 on Standards a new Method of Test for Water-Soluble Acidity or Alkalinity of Paper (D 548 – 39 T) and a Test for Resin in Paper (D 549 – 39 T). These two methods were accepted[1] by Committee E-10 for publication as tentative on August 24, 1939, and appear in the 1939 Book of A.S.T.M. Standards.[2]

NEW TENTATIVE STANDARDS

The committee recommends that the following seven methods of test, which are based upon standard methods of the Technical Association of the Pulp and Paper Industry, be accepted for publication as tentative:

Tentative Methods of:[3]

Sampling Paper and Paper Products,
Test for Casein in Paper (Qualitative),
Test for Alpha-, Beta-, and Gamma-Cellulose in Paper,
Test for Starch in Paper,
Test for Paraffin Content of Waxed Paper,
Test for Opacity of Paper and Paper Products, and
Test for Ash Content of Paper and Paper Products.

These recommendations have been submitted to letter ballot of the committee which consists of 47 voting members; 36

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-6 reported results of the letter ballot vote as follows: Of a total membership of 46, 31 members returned their ballots, of whom 25 voted affirmatively, 0 negatively, and 6 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part III, pp. 1059, 1055.

[3] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 489, 493, 494, 504, 502, 500, and 491, respectively.

members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Standards			
Sampling Paper and Paper Products	26	2	8
Test for Casein in Paper	28	0	8
Test for Alpha, Beta, and Gamma Cellulose in Paper	21	2	13
Test for Starch in Paper	28	0	8
Test for Paraffin Content of Waxed Paper	29	0	7
Test for Opacity of Paper and Paper Products	24	0	12
Test for Ash Content of Paper and Paper Products	29	0	2

Activities of Subcommittees

Subcommittee I on Paper Testing Methods (W. E. Emley, chairman) has held several meetings during the year and has continued its critical study of the various methods of test which have been approved by T.A.P.P.I. The seven methods which are recommended at this time for publication as tentative are the outcome of work done by this subcommittee.

At the present time the following methods are being given consideration by Subcommittee I:

T.A.P.P.I. Standard	Subject
T410m	Basis weight
T412m	Moisture
T414m	Tearing strength
T427m	Saturation properties
T433m	Water resistance
T403m	Bursting strength
T411m	Thickness
T423m	Folding endurance
T424m	Gloss

In addition to these T.A.P.P.I. methods, the subcommittee is also working on test procedures for conditioning, determining tensile strength, and measuring absorption, porosity, stiffness, and curl.

Subcommittee II on Significance of Test Methods (C. C. Heritage, chairman) held a meeting in New York, N. Y., on February 25, 1940, at which the details were worked out for preparing a monograph covering the significance of tests and the definition of terms, nomenclature, and properties of paper, in which the interested committees of A.S.T.M. and T.A.P.P.I. have been invited to cooperate.

Subcommittee IV on Fiberboard and Fiberboard Containers (D. L. Quinn, chairman) has held several meetings throughout the year and has completed the extensive research testing program which was launched last year. A report containing detailed, authoritative, and comprehensive data on the physical properties of container grades of paperboard and fiberboard shipping containers has been compiled and distributed to the members of the subcommittee. The data are to be used as a basis for furthering the subcommittee's program of activities.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Lewis S. Reid.
Vice-Chairman, M. A. Krimmel.
Secretary, G. H. Harnden.

The report of Committee D-6 has been submitted to letter ballot of the committee which consists of 47 voting members; 36 members returned their ballots, of whom 34 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. C. Griffin,
Chairman.

L. S. Reid,
Secretary.

REPORT OF COMMITTEE D-7

ON

TIMBER

Committee D-7 on Timber presents herewith a report of its activities during the past year. The committee has held one formal meeting, in Chicago, Ill., on March 12, 1940.

The committee has only one recommendation to make this year: namely, an editorial change in the Tentative Method of Test for Tar Acids in Creosote and Creosote-Coal Tar Solutions (D 453 - 39 T).[1] Technical Committee XII on Laboratory Apparatus of Committee E-1 on Methods of Testing has recommended that in Figs. 1 and 2 of this method the illustration of the glass separatory funnels be changed to indicate that the tip of the burette shall be 20 mm. long, measured from the outside of the barrel to the end of the ground tip, rather than a distance of 20 mm. from the center of the stopcock to the end of the ground tip. This change will correct an error, which was overlooked in the latest illustrations appearing in the current methods of both the American Wood-Preservers' Association and the Society. It is claimed by the manufacturers that it is practically impossible to make the tips as short as required by the present drawing. Committee D-7 accordingly voted to approve the recommendation made by Committee E-1, and recommends that this change be made when the method is next printed. The method as a whole is being retained as tentative pending further action by committees of the American Railway Engineering Association and the American Wood-Preservers' Association.

The editorial change has been submitted to letter ballot of the committee which consists of 24 members; 23 members returned their ballots, of whom 21 have voted affirmatively, 0 negatively, and 2 members marked their ballots "not voting."

The committee recommends that the Standard Definitions of Terms Relating to Timber Preservatives (D 324 - 33) be continued as standard without change. The definitions have been carefully considered and the committee is unable to suggest any revisions at this time.

As a matter of information, the committee reports that consideration is being given to possible changes in the commercial names for several species of American woods referred to in A.S.T.M. standards. Some of these names do not conform to the rulings of the Federal Trade Commission. The Subcommittee on Nomenclature and Definitions is accordingly engaged in a revision of some of these names, the details of which will be presented next year.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 24 members; 23 members returned their ballots, of whom 22 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

HERMANN VON SCHRENK,
Chairman.

J. A. NEWLIN,
Secretary.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 1126.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

Committee D-8 on Bituminous Waterproofing and Roofing Materials has held two meetings during the past year: in Atlantic City, N. J., on June 28, 1939, and in New York City on April 5, 1940.

Subsequent to the 1939 annual meeting Committee D-8 presented to the Society through Committee E-10 on Standards, a complete revision of the Tentative Specifications for Primer for Use with Asphalt in Dampproofing and Waterproofing Below and Above Ground Level (D 41 – 36 T). The revised specifications were accepted[1] for publication as tentative on August 24, 1939, and appear in the 1939 Book of Standards.[2]

At a meeting on June 27, 1940, the committee referred to letter ballot, a recommendation from its Subcommittee VI on Fiber Analysis (C. S. Reeve, chairman), that the Standard Methods of Analysis of Roofing Felt for Fiber Composition (D 272 – 34) be withdrawn. If the letter ballot is favorable, such recommendation will be made to the Society through Committee E-10 on Standards.[3] The report of Subcommittee VI, together with supporting data bearing upon this method, is appended hereto.

I. REVISION OF TENTATIVE STANDARD

The committee recommends that the Tentative Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 – 39 T)[4] be revised with respect to the ash requirement and continued as tentative. This change is recommended because the present requirement cannot be met by raw cotton fabrics now available, and a more liberal requirement will not affect the quality or serviceability of the product for waterproofing purposes. Accordingly, in Section 3 (*b*) change the maximum requirement for ash (based on dry weight of fabric) from "1 per cent" to read "2 per cent."

II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following two tentative standards be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of:[5]

Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (D 451 – 37 T), and

Test for Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (D 452 – 37 T).

III. REVISION OF STANDARD

At its meeting on June 27, 1940, the committee approved, jointly with Committee D-4 on Road and Paving Materials, the following revision in the Standard Methods of Testing Emulsified

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[1] In submitting these revised tentative specifications to Committee E-10 on Standards, Committee D-8 reported results of the letter ballot vote as follows: Of a voting membership of 59, 34 members returned their ballots, all of whom voted affirmatively.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 1064.

[3] See Editorial Note, p. 347.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 1078.

[5] *Ibid.*, pp. 1101, 1104.

Asphalts (D 244 – 39).[6] This change in the calibration of the centrifuge tube was made on the recommendation of the Section on Volumetric Glassware, of Technical Committee XII, of Committee E-1 on Methods of Testing.

Section 8 (f).—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> The trap shall be made of well-annealed glass constructed in accordance with Fig. 3 (*c*) and shall be graduated from 0 to [25 ml.] *2 ml.* in 0.1-ml. divisions, *and from 2 to 25 ml. in 0.2-ml. divisions.*

IV. TENTATIVE STANDARDS AND REVISIONS CONTINUED AS TENTATIVE

Committee D-8 recommends that the 13 tentative standards and the tentative revisions of two standards listed below be continued as tentative for the reasons indicated:

Tentative Specifications for:

Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (D 43 – 37 T), and

Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (D 450 – 38 T).

Tentative Methods of Testing:

Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 – 38 T),

Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 – 37 T), and

Films Deposited from Bituminous Emulsions (D 466 – 38 T).

These specifications and methods of test are now being reviewed and a number of revisions will probably be recommended next year.

Tentative Specifications for:

Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 – 37 T),

Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 – 37 T),

Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 – 37 T),

Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 – 37 T),

Asphalt Cap Sheet Surfaced with Coarse Mineral Granules (D 371 – 37 T),

Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 – 38 T), and

Asphalt for Dampproofing and Waterproofing (D 449 – 37 T).

Tentative Method of:

Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 – 37 T).

The committee feels that in the first five specifications listed above the requirements for coating and filler weights should be revised and clarified. Consideration is also being given to the inclusion of larger granules for surfacing.

In Specifications D 226 a revision in the weight of finished felt has recently been suggested and until such revision can be given thorough consideration, no change in status of these specifications is recommended.

Final action has been deferred on Specifications D 449 pending an attempt to reach agreement with the American Railway Engineering Association on ductility requirements. That organization now specifies ductility at 40 F. in addition to 77 F., and its requirement of a minimum ductility at 77 F. of 20 cm. as compared with the minimum of 15 cm. for type B material in Specifications D 449 calls for a product which has not been available during the past five years. It is recommended that these specifications be continued as tentative for another year so that a further attempt may be made to settle the points of difference in the specifications of the two organizations.

While it is believed that Method D 313 is ready for adoption as standard, it is so closely related to the Tentative Specifications for Asphalt for Use in

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[6] 1939 Book of A.S.T.M. Standards, Part II, p. 400.

Constructing Built-Up Roof Coverings (D 312 – 39 T) which are now in process of revision that no change in status is recommended at this time.

Tentative Revisions of Standard Specifications for:

Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 227 – 27), and

Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs (D 250 – 27).

Tentative revisions of these two standards were accepted by the Society in 1939. Owing to the fact that further revisions are anticipated in the near future, no change in status is at present recommended.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 60 voting members; 44 members returned their ballots, with the results shown in Table I.

The present officers of the committee were re-elected for the ensuing term of two years.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. Revision of Tentative Standard			
Spec. for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 – 39 T)	37	0	7
II. Adoption of Tentative Standards as Standard			
Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (D 451 - 37 T)	41	0	3
Test for Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (D 452 - 37 T)	40	0	4

This report has been submitted to letter ballot of the committee which consists of 60 voting members; 44 members returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. M. Weiss,
Chairman.

Prévost Hubbard,
Secretary.

Editorial Note

Subsequent to the annual meeting Committee D-8 recommended to the Society through Committee E-10 on Standards the withdrawal of the Standard Methods of Analysis of Roofing Felt for Fiber Composition (D 272 – 34). This recommendation was accepted by Committee E-10 on August 28, 1940.

REPORT OF SUBCOMMITTEE VI ON METHODS OF FIBER ANALYSIS

TABLE I.—COMPOSITIONS OF SAMPLES USED IN COOPERATIVE TESTS AND RESULTS REPORTED BY SEVEN LABORATORIES.

	Composition of sample, per cent	Fiber Analyses Reported by Laboratories, per cent						
		A	B	C	D	E	F	G
Sample No. 1:								
Cotton	40	35.8	40.8	34.5[a]	38	39	35	41.2[b]
Jute	20	9.6	17.0	9.5	15	12	20[b]	11.7
Mechanical Wood	10	5.1[a]	9.2	13.5[b]	13	7	10	11.2
Chemical Wood	10	9.3	13.5	17.8[b]	8[a]	15	15	14.7
Viscose Rayon	20	24.3	19.5	23.0	26[b]	19[a]	20	21.2
Acetate Rayon	0[a]	15.9[b]	0.0[a]	1.7	0[a]	8	0[a]	0.0[a]
Sample No. 2:								
Cotton	40	23.0[a]	39.3	33.7	41	41	45[b]	38.2
Jute	20	6.2[a]	23.8[b]	13.1	20	19	20	19.2
Mechanical Wood	10	12.2	10.4	16.0	10[a]	13	10[a]	16.5[b]
Chemical Wood	10	11.7[a]	15.5	16.8[b]	13	20	15	16.2
Acetate Rayon	20	27.6[b]	11.0	14.0	16	3[a]	10	8.6
Viscose Rayon	0[a]	19.3[b]	0.0[a]	6.4	0[a]	4	0[a]	1.3
Sample No. 3:								
Cotton	30	20.4[a]	33.9	32.8	38[b]	34	35	30.9
Jute	30	12.1[a]	27.0[b]	14.6	19	21	25	19.4
Mechanical Wood	10	11.9	10.2	18.5	13	12	10[a]	19.8[b]
Chemical Wood	10	10.1[a]	13.4	14.7	11	15[b]	15[b]	12.6
Viscose Rayon	10	22.9[b]	13.1	15.8	14	13	10[a]	13.8
Acetate Rayon	10	22.6[b]	2.4[a]	3.6	5	5	5	3.5

[a] Low.

[b] High.

In view of the increasing rayon content of rags, the subcommittee was requested to investigate the possibility of extending the Standard Methods of Analysis of Roofing Felt for Fiber Composition (D 272 – 34)[1] to include the determination of rayon fibers. As a result of correspondence within the subcommittee and independent laboratory tests, it was decided for the purpose of making cooperative tests to supplement the present procedure as indicated below:

Proposed Method of Test for Rayon in Roofing Felt:

The A.S.T.M. Standard Methods of Analysis of Roofing Felt for Fiber Composition (D 272 – 34)[1] shall be used, except that rayon shall be determined in addition to those fibers listed in the method.

In order to identify rayon fibers it is recommended that the operator prepare slides of viscose and acetate rayon fibers (furnished to the cooperators) and, using Herzberg stain, observe the fibers for color reaction and structure, which may be described as follows:

Viscose and Cuprammonium Rayon.—

COLOR REACTION: Lavender to purple or black. The light-colored rayon stains lavender but changes rapidly to purple. The dark-colored rayon stains purple or black immediately.

STRUCTURE: Cylindrical smooth fiber walls comparatively uniform in diameter.

Acetate Rayon.—COLOR REACTION: Brown or yellow. Black dyed rayon reacts slowly.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 484.

STRUCTURE: The fibers swell rapidly and become shapeless. In some cases the dye is retained in the fiber canal and is visible after the fiber wall has dissolved.

After the count has been made and if acetate rayon was detected, it is recommended that freshly stained slides be examined rapidly and the acetate rayon approximated as a check on the acetate rayon count.

In order to determine the possible agreement between different operators using this method, handmade sheets of known composition were carefully prepared. These contained one or both types of rayon in combination with cotton, jute, and chemical and mechanical wood. The sheets were distributed among the seven laboratories, which expressed a desire to cooperate. Included with the test sheets were samples of each of the types of rayon to enable the operator to become familiar with their characteristic behavior before attempting to make the analysis.

The results reported by the seven laboratories in comparison with the known composition of the samples are presented in Table I.

The results were discouraging, not only in failing to disclose a satisfactory method for determining rayon but also in showing the inaccuracy of the method for determining other common fibers when rayon is present. It is also appreciated that the matter will be further complicated with the inclusion of the newer synthetic chemical fibers in rags. The test is not required in any A.S.T.M. specifications coming under the jurisdiction of Committee D-8, and in view of its apparent inadequacy the subcommittee voted unanimously for the withdrawal of the present method from the Book of Standards.[2]

Respectfully submitted on behalf of the subcommittee,

C. S. REEVE,
Chairman.

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[2] See Editorial Note, p. 347.

REPORT OF COMMITTEE D-9

ON

ELECTRICAL INSULATING MATERIALS

Committee D-9 on Electrical Insulating Materials held two meetings during the year and will hold a third during the annual meeting of the Society in June at Atlantic City, N. J. The meetings were held in October, 1939, and February, 1940, with an average attendance of 82 members and 13 visitors. During the year considerable progress was made in the work which was pending and several projects have reached the stage where definite recommendations have been made, resulting in additions or changes in the standards for which Committee D-9 is responsible. Other projects which are in progress are mentioned later in this report.

Subsequent to the 1939 annual meeting, Committee D-9 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of:

Testing Glass Spool Insulators (D 550 - 39 T),

Measuring Shrinkage from Mold Dimensions of Molded Materials Used for Electrical Insulation (D 551 - 39 T),

Revisions of Tentative Methods of:

Testing Varnishes Used for Electrical Insulation (D 115 - 38 T),

Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 36 T),

Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 38 T),

Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 38 T),

Tentative Revisions of Standard Methods of:

Testing Pin-Type, Lime Glass Insulators (D 468 - 39 T),

Testing Molded Materials Used for Electrical Insulation (D 48 - 39), and

Test for Grading and Classification of Natural Mica (D 351 - 38) in the form of a new Tentative Method of Test for Power Factor and Dielectric Constant of Natural Mica (D 351 - 39 T), to be added when adopted to the present standard.

These recommendations were accepted[1] by Committee E-10 on October 7, 1939; the new and revised tentative methods and tentative revisions of standards appear in the 1939 Book of A.S.T.M. Standards.[2]

RECOMMENDATIONS AFFECTING STANDARDS

As a result of the year's work, the committee is submitting revisions of four tentative standards, tentative revisions of five existing standards, and is recommending the adoption as standard of two tentative standards and one tentative revision of an existing standard. The recommendations being presented are included in detail in the Appendix, each proposed revision being accompanied by a brief explanation of the reason for the revision. The standards affected are listed in Table I.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-9 reported the following results of the letter ballot vote of a total of 49 ballots returned from a committee membership of 90: D 550 - 39 T, affirmative 18, negative 0, ballots marked "not voting" 31; D 551 - 39 T, affirmative 16, negative 0, ballots marked "not voting" 33; D 115 - 38 T, affirmative 21, negative 0. ballots marked "not voting" 28; D 150 - 36 T, affirmative 35, negative 0, ballots marked "not voting" 14; D 202 - 38 T, affirmative 24, negative 0, ballots marked "not voting" 25; D 350 - 38 T, affirmative 21, negative 0, ballots marked "not voting" 28: D 468 - 39 T, affirmative 17, negative 1, ballots marked "not voting" 31; D 48 - 39, affirmative 23, negative 0, ballots marked "not voting" 26; D 351 - 38, affirmative 22, negative 0, ballots marked "not voting" 27.

[2] 1939 Book of A.S.T.M. Standards, Part III, pp. 719, 698, 662, 745, 780, 686, 711, 1138, 800, respectively.

These recommendations have been submitted to letter ballot of the committee which consists of 92 voting members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Since a large part of the subcommittee work consists of obtaining data and studying results with a view to improving the present standards and developing new standards, it is only possible in one year to submit definite recommendations on a portion of the work. Some of the projects that are in progress are as follows:

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISIONS OF TENTATIVE STANDARDS			
Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 39 T)	33	1	40
Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 39 T)	23	0	29
Spec. for Flexible Varnished Tubing Used in Electrical Insulation (D 372 37 T)	23	0	29
Spec. for Phenolic Laminated Sheet for Radio Applications (D 467 - 37 T)	23	0	29
II. TENTATIVE REVISIONS OF STANDARDS			
Testing Molded Materials Used for Electrical Insulation (D 48 - 39)	34	0	40
Testing Electrical Insulating Oils (D 117 - 36)	28	0	24
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 39):			
Revision of water absorption test (Sections 19 to 28)	32	0	20
Revision of hardness test (Sections 41 and 42)	37	1	36
Testing Laminated Tubes Used in Electrical Insulation (D 348 - 39)	30	0	22
Testing Laminated Round Rods Used in Electrical Insulation (D 349 - 39)	29	0	23
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Conducting Paths in Electrical Slate (D 273 - 36 T)	29	0	23
Testing Shellac Used for Electrical Insulation (D 411 - 37 T)	18	0	34
IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Testing Electrical Insulating Oils (D 117 - 36)	26	0	26

A Joint Subcommittee on Water Absorption, composed of representatives of Subcommittee V of Committee D-20 on Plastics and Subcommittees II and III of Committee D-9, has reviewed the proposed Method of Test for Water Absorption of Plastics, prepared by Committee D-20, and has agreed to various changes to permit the same method of test to be used by both committees. Upon the recommendation of the Joint Subcommittee, Subcommittees II and III submitted the method to letter ballot for approval, with favorable action. The proposed method is being presented to the Society by Committee D-20 for publication as tentative at this annual meeting.[3] This method upon acceptance is intended to replace the test for water absorption described in the Standard Methods of Testing Molded Materials (D 48 – 39), Laminated Tubes (D 348 – 39), Laminated Round Rods (D 349 – 39), and method *A* for rate of absorption in the Standard Methods of Testing Sheet and Plate Materials (D 229 – 39) Used in Electrical Insulation.

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (C. F. Hanson, chairman).—The British Draft Standard Specifications for Lac (CF (B) 2368) have been carefully reviewed and a complete report has been transmitted to Sectional Committee C59 on Electrical Insulating Materials in order that it may pass through the proper channels for consideration by the International Electrotechnical Commission and the British Standards Institution.

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[3] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 292.

Considerable progress has been made in an effort to develop a suitable test method for determining the resistance of varnish films to alkali solutions, sulfuric acid solutions, and synthetic sea water. Five laboratories are participating actively.

This subcommittee is cooperating in the effort to standardize baking ovens and drying cabinets for use in determining the drying time of baking varnishes and air drying varnishes. Work is still in progress to develop a method of testing the internal drying time of saturant varnishes. Attention is also being given to a method of testing the set time of phenolic resin varnishes.

Subcommittee II on Molded Insulating Materials (W. A. Evans, chairman).—A standard mold is being developed for compression-molded tension specimens. A round-robin series of tests will be conducted in connection with this development.

Work on conditioning test specimens of thermal plastics is being continued in cooperation with Subcommittee X.

A new section was formed to review the Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 39). It has recommended various ways of simplification.

Draft specifications for phenolic molded materials used for electrical insulation have been presented by the chairman and a section is to be appointed to carry forward the work on such specifications.

The Tentative Method of Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 – 38 T) is being continued as tentative; however, it will probably be considered for adoption as standard at the next meeting of the committee.

Subcommittee III on Plates, Tubes, and Rods (Dean Harvey, chairman).—Methods for measuring dimensions of laminated, stiff tubing are being prepared. A method for determining the dielectric strength of laminated tubing parallel to laminations is being developed, and an impact test for rods and tubing to determine the effect of riveting operations is under consideration.

Methods of conditioning sheet, rod, and tube materials to insure reproducibility of electrical tests for power factor, insulation resistance, and dielectric strength are being developed, and comparative tests are being made to obtain the necessary information.

A very comprehensive series of tests comparing actual punching of laminated sheets by means of a die with Rockwell hardness tests at elevated temperatures has shown that a definite relation exists for certain materials which will permit the Rockwell test to be used as an indication of punching quality. The method of test is being prepared.

Further work is being done to determine which tests are most suitable for checking the uniformity of phenolic laminated sheets. The work on paper-base sheets is nearly complete, and tests are being started on fabric-base sheets.

The proposed test for power factor of phenolic laminated sheets parallel to laminations is still under consideration. A test for ply adhesion of vulcanized fiber is being developed.

Subcommittee IV on Liquid Insulation (E. A. Snyder, chairman).—Work has been continued during the year in cooperation with Committee D-2 on Petroleum Products to improve the test for neutralization number (D 188 – 27 T)[4] and to develop an electrometric titration method. A year ago it was felt that a suitable electrometric titration method could be prepared; however, the great amount of work done by both Committees D-9 and D-2 during the past year has brought out the fact that none of the proposed methods is free from objectionable criticism and, if anything, the development of a satisfactory test appears further away today than a year ago. Work is being continued on this subject in both committees.

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[4] See pp. 301 and 309.

The results of the year's study on the development of a sludge accumulation test and a high-pressure bomb test indicate that steady progress has been made in the sludge accumulation test and considerable promise of success with the high-pressure bomb test. A second series of oils is undergoing a round-robin test at present and if the results on this series are as good as those obtained on the first series, a tentative sludge accumulation test may be presented during the coming year.

A canvass of the field of users and producers of insulating oils indicated that an evaporation test is not significant as a criterion of insulating oils and is not needed by the operators of transformers and switches. Further study is to be held in abeyance and the work will not be revived unless there is a definite demand and need expressed for it.

Work is being continued with Subcommittee VII in the development of a suitable cell for determining power factor of oils; however, trouble has been encountered in securing a cell which will give consistent check test results.

After a great amount of work a modification of the test for dielectric strength has been completed and is being presented as a tentative revision[5] of the Standard Methods of Testing Electrical Insulating Oils (D 117 - 40).

Probably one of the most important developments of the subcommittee has been the formation of a section to study the question of minute traces of moisture in insulating oils. This section will study the significance of minute traces of moisture in relation to service of transformers, switches, etc., and it will investigate the various proposed methods of determining minute traces of moisture in an attempt to develop a satisfactory method of test.

Sections are also active in the following subjects: (*1*) a study of the gas content of oil and the development of methods of measuring it, (*2*) the development of statements regarding the significance of the tests described in Methods D 117 - 36 as applied to insulating oils, and (*3*) a study of the applicability of the tests covered by Methods D 117 - 36 to insulating mediums other than insulating oil, that is, synthetic insulating liquids, and the preparation of new methods where the present ones are not suitable.

Subcommittee IV is continuing to keep in close contact with the International Electrotechnical Commission, the chairman acting as technical advisor on insulating oils to the U. S. National Committee of the I. E. C. During the past year F. M. Clark was able personally to contact many members of Committee No. 10 of the I. E. C. while visiting in various foreign countries, and, as a result, closer working arrangements have been set up with H. W. Bruckman of Delft, Holland, who is acting as secretary of Committee No. 10. Samples of oil and test methods are being exchanged at present.

Subcommittee V on Ceramic Products (Porcelain, Glass, Etc.) (L. E. Barringer, chairman).—There has been no change in the status of the porosity test by the dye penetration method as the American Institute of Electrical Engineers has not as yet taken final action on the revision of the A. I. E. E. standard in which the dye penetration method is included.

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[5] See p. 359.

It has been generally agreed that the present Standard Methods of Testing Electrical Porcelain (D 116 – 39)[6] are suitable for testing steatite but should be modified to permit the testing of smaller size specimens for mechanical strength. In order to establish the correct test specimens it was agreed that a series of tests be made in several laboratories. Tests will be made for flexual strength, tensile strength, and resistance to impact.

Test data are to be assembled on the method of determining water absorption of porcelain.

It has been proposed to invite representatives of several other societies to work with the Section on Glass Insulators in the design of a standard thread gage for pin type glass insulators. Several gages have been made and are being checked by different laboratories. In view of the activities under way in other societies it has been decided to hold in abeyance the preparation of methods of testing low-expansion glass insulators.

Subcommittee VI on Solid Filling, and Treating Compound (R. H. Titley, chairman).—The Tentative Methods of Testing Solid Filling and Treating Compounds (D 176 – 37 T) are being continued as tentative[6] pending the results of further tests. Interest in this field has lessened somewhat in recent years. However, with the advent of the newer compounds for filling joints and terminals, interest in methods of testing these compounds is being revived, particularly as to their solubility in cable oil. Studies will be made by this subcommittee in the near future of tests applicable to the newer compounds.

Subcommittee VII on Electrical Tests (J. A. Scott, chairman).—Work in progress on the measurement of the power factor of mineral insulating oil indicates that bridge circuit agreement approaching 10^{-5} in power factor can be obtained, but that differences many times as great occur in actual measurements on oil.

The Tentative Method of Test for Comparing Thermal Conductivities of Solid Electrical Insulating Materials (D 325 – 31 T) is being continued as tentative. Informal discussion with Committee C-16 on Thermal Insulating Materials may broaden the usefulness of the method or result in a revision of it.

The Tentative Methods of Test for Conducting Paths in Electrical Slate (D 273 – 36 T), having being published as tentative for four years without revision or criticism, are being recommended for adoption as standard. These methods are not widely used because of the decreasing use of slate as a switchboard material. However, considerable time and effort were expended in their preparation and it is therefore the feeling of the committee that they should be continued as an A.S.T.M. standard, but that publication of the methods repeatedly may not be necessary.

The study of power factor measurements of electrical insulating materials at frequencies up to 100 megacycles has been continued actively and promises to result in the development of a satisfactory method that may be recommended for publication next year.

Subcommittee VIII on Insulating Papers and Fabrics (Treated and Untreated) (R. W. Chadbourn, chairman).—The subcommittee is continuing its investigation of the pH method for determining the acidity of untreated papers, employed by H. F. Launer of the National Bureau of Standards. In connection with these studies several questions have arisen. It is felt that a pH test, supplemented by a conductivity test, is a more satisfactory control test than a determination of total solids by evaporation to dryness. Also, in regard to time and temperature of extraction, the requirements for somewhat alkaline papers may be different from those of highly acidic papers. In the case of strongly acidic papers, the error due to the generation of weak acids during extraction would be inappreciable; in the case of less acidic, or of moderately alkaline papers, this might not be the case.

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[6] **See Editorial Note, p. 356.**

Further progress was reported in the development by Committee D-6 on Paper and Paper Products of methods of mechanical tests of papers. The standard for bursting strength, involving both precision and control methods, was reported already acceptable to Committee D-6 and the Technical Association of the Pulp and Paper Industry. Progress has been made with respect to the tensile strength, folding endurance, and stiffness test methods. It is expected that the method of test for folding endurance will include both the M.I.T. and Schopper testers. No decision has been reached on edge-tear testers.

The comparative laboratory tests for thickness measurements of paper, involving three types of micrometers (standard machinist's, machinist's with adjusted pressure, and dead-weight dial gage) are still in progress. The Tentative Methods of Test for Thickness of Solid Electrical Insulation (D 374 - 36 T) are being continued as tentative until completion of these tests.

Special samples of varnished cloth tape, embodying various stretch characteristics, have been distributed for test to a number of laboratories of utility companies. The Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 38 T) are being continued as tentative pending the results of the stretch test.

Procedures for testing saturated sleeving and the addition of larger sizes of tubing are being incorporated in the Tentative Methods of Testing Flexible Varnished Tubing (D 350 - 39 T).

Results of a questionnaire indicated sufficient interest in cellulose acetate and woven glass tapes to warrant consideration of these materials at this time. It has been pointed out that there are two distinct grades of woven glass tape (untreated and treated) which might require more or less different testing procedures, and some doubt has been expressed as to whether the impregnation of tapes had been sufficiently standardized to warrant consideration of the treated type at present. Due to the work on glass tapes by Committee D-13 on Textile Materials, it was agreed that whatever work might be undertaken by Subcommittee VIII should be confined to a study of electrical characteristics and should supplement the work being done by Committee D-13.

Subcommittee IX on Mica Products (M. P. Davis, chairman).—Methods of measuring mica stampings used in radio tubes and incandescent lamps are being prepared. The grading chart for mica in the Standard Methods of Test for Grading and Classification of Natural Mica (D 351 - 38) is to be revised to show a grade No. 4½, and grade No. 6 is to be subdivided into two or three grades. The preparation of color photographs or color transparencies for classification of mica is being continued. Work is also being continued on the preparation of purchase specifications for mica and mica products.

Subcommittee X on Conditioning (Robert Burns, chairman).—This subcommittee is carrying on cooperative work with other subcommittees of Committee D-9 and with other standing committees of the Society, as for example, in the development of conditioning methods for the mechanical strength tests being considered by Committee D-20 on Plastics.

Work is also in progress on the establishment of definitions for combinations of atmospheric influences, and in the design of standardized testing equipment.

Special Subcommittee on Significance of Tests (Dean Harvey, chairman).—This subcommittee is cooperating with special groups in each subcommittee in preparing reports on the significance of the tests covered by the respective subcommittees.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 92 voting members; 52 members returned their ballots, of whom 45 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. SMITH TAYLOR,
Chairman.

E. J. RUTAN,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-9 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Methods of:

- Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 - 39 T),
- Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 37 T),
- Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 40 T),

Tentative Revision of Standard Methods of:

- Testing Molded Materials Used for Electrical Insulation (D 48 - 39), and
- Testing Electrical Porcelain (D 116 - 39).

These recommendations were accepted by Committee E-10 on August 28, 1940, and the tentative methods in their revised form and the tentative revisions appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 257, 242, 264, 563, and 569, respectively.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentative standards covering electrical insulating materials which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the standard appears in its present form.

Revisions of Tentative Standards

Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 39 T):[1, 2]

Changes in the survey for conducting paths described at present in Sections 51 to 55, inclusive of the methods are recommended. Since these changes are rather extensive the revised sections designated 51 to 64 are appended hereto,[3] the remaining sections to be renumbered accordingly. This revision also replaces the present appendix to the methods.

Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 39 T):[4]

It is recommended that the scope of these methods be enlarged to include saturated sleeving. Also, to meet the demand for larger sizes of tubing than those now standardized, a number of additional sizes, ranging up to 1 in., in inside diameter are proposed. Experience has shown that some of the permissible variations in the smaller sizes are inadequate; a slight increase in limits for these sizes is recommended.

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> These methods cover the procedures for *the* testing *of* flexible varnished tubing *and saturated sleeving* (Note) used as electrical insulation for leads on electrical apparatus.
>
> Note.—The term "varnished tubing" as [here] used *in these methods* refers to braided cotton sleeving coated, or impregnated and coated, with *varnish*, lacquer, or a combination of lacquer and varnish, or [other] similar coatings. *The term "saturated sleeving" refers to braided cotton sleeving impregnated with varnish but the coating need not be continuous as it is for varnished tubing.*

Table I.—Add seven new sizes of tubing at the beginning of the table to read as follows:

Size	Inside Diameter, in.	
	Maximum	Minimum
1 in.	1.036	1.000
7/8 in.	0.911	0.875
3/4 in.	0.786	0.750
5/8 in.	0.655	0.625
1/2 in.	0.524	0.500
7/16 in.	0.462	0.438
3/8 in.	0.399	0.375

Change the maximum inside diameters for sizes Nos. 14 and smaller to read as follows by the addition of the

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[1] 1939 Book of A.S.T.M. Standards, Part III, p. 780.

subsequent to the annual meeting, see Editorial Note and a complete report has been transmitted to Sectional Committee C59 on Electrical Insulating Materials in order that it may pass through the proper channels for consideration by the International Electrotechnical Commission and the British Standards Institution.

Considerable progress has been made in an effort to develop a suitable test method for determining the resistance of varnish films to alkali solutions, sulfuric acid solutions, and synthetic sea water. Five laboratories are participating actively.

This subcommittee is cooperating in the effort to standardize baking ovens and drying cabinets for use in determining the drying time of baking varnishes and air drying varnishes. Work is still in progress to develop a method of testing the internal drying time of saturant varnishes. Attention is also being given to a method of testing the set time of phenolic resin varnishes.

Subcommittee II on Molded Insulating Materials (W. A. Evans, chairman).—A standard mold is being developed for compression-molded tension specimens. A round-robin series of tests will be conducted in connection with this development.

Work on conditioning test specimens of thermal plastics is being continued in cooperation with Subcommittee X.

A new section was formed to review the Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 39). It has recommended various ways of simplification.

Draft specifications for phenolic molded materials used for electrical insulation have been presented by the chairman and a section is to be appointed to carry forward the work on such specifications.

The Tentative Method of Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 – 38 T) is being continued as tentative; however, it will probably be considered for adoption as standard at the next meeting of the committee.

Subcommittee III on Plates, Tubes, and Rods (Dean Harvey, chairman).—Methods for measuring dimensions of laminated, stiff tubing are being prepared. A method for determining the dielectric strength of laminated tubing parallel to laminations is being developed, and an impact test for rods and tubing to determine the effect of riveting operations is under consideration.

Methods of conditioning sheet, rod, and tube materials to insure reproducibility of electrical tests for power factor, insulation resistance, and dielectric strength are being developed, and comparative tests are being made to obtain the necessary information.

A very comprehensive series of tests comparing actual punching of laminated sheets by means of a die with Rockwell hardness tests at elevated temperatures has shown that a definite relation exists for certain materials which will permit the Rockwell test to be used as an indication of punching quality. The method of test is being prepared.

Further work is being done to determine which tests are most suitable for checking the uniformity of phenolic laminated sheets. The work on paper-base sheets is nearly complete, and tests are being started on fabric-base sheets.

The proposed test for power factor of phenolic laminated sheets parallel to laminations is still under consideration. A test for ply adhesion of vulcanized fiber is being developed.

Subcommittee IV on Liquid Insulation (E. A. Snyder, chairman).—Work has been continued during the year in cooperation with Committee D-2 on Petroleum Products to improve the test for neutralization number (D 188 – 27 T)[4] and to develop an electrometric titration method. A year ago it was felt that a suitable electrometric titration method could be prepared; however, the great amount of work done by both Committees D-9 and D-2 during the past year has brought out the fact that none of the proposed methods is free from objectionable criticism and, if anything, the development of a satisfactory test appears further away today than a year ago. Work is being continued on this subject in both committees.

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[4] See pp. 301 and 309.

The results of the year's study on the development of a sludge accumulation test and a high-pressure bomb test indicate that steady progress has been made in the sludge accumulation test and considerable promise of success with the high-pressure bomb test. A second series of oils is undergoing a round-robin test at present and if the results on this series are as good as those obtained on the first series, a tentative sludge accumulation test may be presented during the coming year.

A canvass of the field of users and producers of insulating oils indicated that an evaporation test is not significant as a criterion of insulating oils and is not needed by the operators of transformers and switches. Further study is to be held in abeyance and the work will not be revived unless there is a definite demand and need expressed for it.

Work is being continued with Subcommittee VII in the development of a suitable cell for determining power factor of oils; however, trouble has been encountered in securing a cell which will give consistent check test results.

After a great amount of work a modification of the test for dielectric strength has been completed and is being presented as a tentative revision[5] of the Standard Methods of Testing Electrical Insulating Oils (D 117 – 40).

Probably one of the most important developments of the subcommittee has been the formation of a section to study the question of minute traces of moisture in insulating oils. This section will study the significance of minute traces of moisture in relation to service of transformers, switches, etc., and it will investigate the various proposed methods of determining minute traces of moisture in an attempt to develop a satisfactory method of test.

Sections are also active in the following subjects: (*1*) a study of the gas content of oil and the development of methods of measuring it, (*2*) the development of statements regarding the significance of the tests described in Methods D 117 – 36 as applied to insulating oils, and (*3*) a study of the applicability of the tests covered by Methods D 117 – 36 to insulating mediums other than insulating oil, that is, synthetic insulating liquids, and the preparation of new methods where the present ones are not suitable.

Subcommittee IV is continuing to keep in close contact with the International Electrotechnical Commission, the chairman acting as technical advisor on insulating oils to the U. S. National Committee of the I. E. C. During the past year F. M. Clark was able personally to contact many members of Committee No. 10 of the I. E. C. while visiting in various foreign countries, and, as a result, closer working arrangements have been set up with H. W. Bruckman of Delft, Holland, who is acting as secretary of Committee No. 10. Samples of oil and test methods are being exchanged at present.

Subcommittee V on Ceramic Products (Porcelain, Glass, Etc.) (L. E. Barringer, chairman).—There has been no change in the status of the porosity test by the dye penetration method as the American Institute of Electrical Engineers has not as yet taken final action on the revision of the A. I. E. E. standard in which the dye penetration method is included.

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[5] See p. 359.

It has been generally agreed that the present Standard Methods of Testing Electrical Porcelain (D 116 – 39)[6] are suitable for testing steatite but should be modified to permit the testing of smaller size specimens for mechanical strength. In order to establish the correct test specimens it was agreed that a series of tests be made in several laboratories. Tests will be made for flexual strength, tensile strength, and resistance to impact.

Test data are to be assembled on the method of determining water absorption of porcelain.

It has been proposed to invite representatives of several other societies to work with the Section on Glass Insulators in the design of a standard thread gage for pin type glass insulators. Several gages have been made and are being checked by different laboratories. In view of the activities under way in other societies it has been decided to hold in abeyance the preparation of methods of testing low-expansion glass insulators.

Subcommittee VI on Solid Filling, and Treating Compound (R. H. Titley, chairman).—The Tentative Methods of Testing Solid Filling and Treating Compounds (D 176 – 37 T) are being continued as tentative[6] pending the results of further tests. Interest in this field has lessened somewhat in recent years. However, with the advent of the newer compounds for filling joints and terminals, interest in methods of testing these compounds is being revived, particularly as to their solubility in cable oil. Studies will be made by this subcommittee in the near future of tests applicable to the newer compounds.

Subcommittee VII on Electrical Tests (J. A. Scott, chairman).—Work in progress on the measurement of the power factor of mineral insulating oil indicates that bridge circuit agreement approaching 10^{-5} in power factor can be obtained, but that differences many times as great occur in actual measurements on oil.

The Tentative Method of Test for Comparing Thermal Conductivities of Solid Electrical Insulating Materials (D 325 – 31 T) is being continued as tentative. Informal discussion with Committee C-16 on Thermal Insulating Materials may broaden the usefulness of the method or result in a revision of it.

The Tentative Methods of Test for Conducting Paths in Electrical Slate (D 273 – 36 T), having being published as tentative for four years without revision or criticism, are being recommended for adoption as standard. These methods are not widely used because of the decreasing use of slate as a switchboard material. However, considerable time and effort were expended in their preparation and it is therefore the feeling of the committee that they should be continued as an A.S.T.M. standard, but that publication of the methods repeatedly may not be necessary.

The study of power factor measurements of electrical insulating materials at frequencies up to 100 megacycles has been continued actively and promises to result in the development of a satisfactory method that may be recommended for publication next year.

Subcommittee VIII on Insulating Papers and Fabrics (Treated and Untreated) (R. W. Chadbourn, chairman).—The subcommittee is continuing its investigation of the pH method for determining the acidity of untreated papers, employed by H. F. Launer of the National Bureau of Standards. In connection with these studies several questions have arisen. It is felt that a pH test, supplemented by a conductivity test, is a more satisfactory control test than a determination of total solids by evaporation to dryness. Also, in regard to time and temperature of extraction, the requirements for somewhat alkaline papers may be different from those of highly acidic papers. In the case of strongly acidic papers, the error due to the generation of weak acids during extraction would be inappreciable; in the case of less acidic, or of moderately alkaline papers, this might not be the case.

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[6] See Editorial Note, p. 356.

Further progress was reported in the development by Committee D-6 on Paper and Paper Products of methods of mechanical tests of papers. The standard for bursting strength, involving both precision and control methods, was reported already acceptable to Committee D-6 and the Technical Association of the Pulp and Paper Industry. Progress has been made with respect to the tensile strength, folding endurance, and stiffness test methods. It is expected that the method of test for folding endurance will include both the M.I.T. and Schopper testers. No decision has been reached on edge-tear testers.

The comparative laboratory tests for thickness measurements of paper, involving three types of micrometers (standard machinist's, machinist's with adjusted pressure, and dead-weight dial gage) are still in progress. The Tentative Methods of Test for Thickness of Solid Electrical Insulation (D 374 - 36 T) are being continued as tentative until completion of these tests.

Special samples of varnished cloth tape, embodying various stretch characteristics, have been distributed for test to a number of laboratories of utility companies. The Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 38 T) are being continued as tentative pending the results of the stretch test.

Procedures for testing saturated sleeving and the addition of larger sizes of tubing are being incorporated in the Tentative Methods of Testing Flexible Varnished Tubing (D 350 - 39 T).

Results of a questionnaire indicated sufficient interest in cellulose acetate and woven glass tapes to warrant consideration of these materials at this time. It has been pointed out that there are two distinct grades of woven glass tape (untreated and treated) which might require more or less different testing procedures, and some doubt has been expressed as to whether the impregnation of tapes had been sufficiently standardized to warrant consideration of the treated type at present. Due to the work on glass tapes by Committee D-13 on Textile Materials, it was agreed that whatever work might be undertaken by Subcommittee VIII should be confined to a study of electrical characteristics and should supplement the work being done by Committee D-13.

Subcommittee IX on Mica Products (M. P. Davis, chairman).—Methods of measuring mica stampings used in radio tubes and incandescent lamps are being prepared. The grading chart for mica in the Standard Methods of Test for Grading and Classification of Natural Mica (D 351 - 38) is to be revised to show a grade No. 4½, and grade No. 6 is to be subdivided into two or three grades. The preparation of color photographs or color transparencies for classification of mica is being continued. Work is also being continued on the preparation of purchase specifications for mica and mica products.

Subcommittee X on Conditioning (Robert Burns, chairman).—This subcommittee is carrying on cooperative work with other subcommittees of Committee D-9 and with other standing committees of the Society, as for example, in the development of conditioning methods for the mechanical strength tests being considered by Committee D-20 on Plastics.

Work is also in progress on the establishment of definitions for combinations of atmospheric influences, and in the design of standardized testing equipment.

Special Subcommittee on Significance of Tests (Dean Harvey, chairman).—This subcommittee is cooperating with special groups in each subcommittee in preparing reports on the significance of the tests covered by the respective subcommittees.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 92 voting members; 52 members returned their ballots, of whom 45 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. SMITH TAYLOR,
Chairman.

E. J. RUTAN,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-9 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Methods of:

Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 - 39 T),

Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 37 T),

Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 40 T),

Tentative Revision of Standard Methods of:

Testing Molded Materials Used for Electrical Insulation (D 48 - 39), and

Testing Electrical Porcelain (D 116 - 39).

These recommendations were accepted by Committee E-10 on August 28, 1940, and the tentative methods in their revised form and the tentative revisions appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 257, 242, 264, 563, and 569, respectively.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentative standards covering electrical insulating materials which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the standard appears in its present form.

REVISIONS OF TENTATIVE STANDARDS

Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 39 T):[1, 2]

Changes in the survey for conducting paths described at present in Sections 51 to 55, inclusive of the methods are recommended. Since these changes are rather extensive the revised sections designated 51 to 64 are appended hereto,[3] the remaining sections to be renumbered accordingly. This revision also replaces the present appendix to the methods.

Tentative Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 – 39 T):[4]

It is recommended that the scope of these methods be enlarged to include saturated sleeving. Also, to meet the demand for larger sizes of tubing than those now standardized, a number of additional sizes, ranging up to 1 in., in inside diameter are proposed. Experience has shown that some of the permissible variations in the smaller sizes are inadequate; a slight increase in limits for these sizes is recommended.

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> These methods cover the procedures for *the* testing *of* flexible varnished tubing *and saturated sleeving* (Note) used as electrical insulation for leads on electrical apparatus.
>
> NOTE.—The term "varnished tubing" as [here] used *in these methods* refers to braided cotton sleeving coated, or impregnated and coated, with *varnish,* lacquer, or a combination of lacquer and varnish, or [other] similar coatings. *The term "saturated sleeving" refers to braided cotton sleeving impregnated with varnish but the coating need not be continuous as it is for varnished tubing.*

Table I.—Add seven new sizes of tubing at the beginning of the table to read as follows:

Size	Inside Diameter, in. Maximum	Inside Diameter, in. Minimum
1 in.	1.036	1.000
7/8 in.	0.911	0.875
3/4 in.	0.786	0.750
5/8 in.	0.655	0.625
1/2 in.	0.524	0.500
7/16 in.	0.462	0.438
3/8 in.	0.399	0.375

Change the maximum inside diameters for sizes Nos. 14 and smaller to read as follows by the addition of the italicized figures and the omission of those in brackets, the values for sizes Nos. 0 to 13 inclusive, to remain unchanged.

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[1] 1939 Book of A.S.T.M. Standards, Part III, p. 780.

[2] A further revision in these methods was submitted subsequent to the annual meeting, see Editorial Note, p. 356.

[3] This revision was accepted by the Society and has been incorporated in the methods which appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 264.

[4] 1939 Book of A.S.T.M. Standards, Part III, p. 686.

No. 14	[0.070] *0.072*
No. 15	[0.063] *0.067*
No. 16	[0.055] *0.061*
No. 17	[0.049] *0.054*
No. 18	[0.044] *0.049*
No. 19	[0.040] *0.044*
No. 20	[0.036] *0.039*

Designate the note at the end of Table I as Note 1 and change to read as follows by the addition of the italicized words:

NOTE 1.—The minimum inside diameters *prescribed* in this table *for sizes Nos. 0 to 20, inclusive,* are the same as the diameters of copper wire for corresponding A.w.g. or B. & S. gage sizes.

Add a new note at the end of Table I to read as follows:

NOTE 2.—Sizes Nos. 14, 16, 18, and 20 are standard and should be specified if possible. Sizes Nos. 15, 17, and 19 are special and may eventually be eliminated.

Section 14 (a).—Change the table to read as follows by the addition of the italicized figures:

Size	Diameter of Test Rod, in.
1 to $\frac{3}{4}$ in., incl	$\frac{7}{16}$
$\frac{5}{8}$ to $\frac{3}{8}$ in., incl	$\frac{3}{8}$
Nos. 0 to 5 incl	$\frac{5}{16}$
Nos. 6 to 11, incl	$\frac{1}{4}$
Nos. 12 and smaller	$\frac{1}{8}$

Tentative Specifications for Flexible Varnished Tubing Used in Electrical Insulation (D 372 - 37 T):[5]

It is also recommended that the scope of these methods be enlarged to include saturated sleeving for the reasons indicated above for similar changes in specifications D 350. The specifications in their revised form are appended hereto.[6]

Tentative Specifications for Phenolic Laminated Sheet for Radio Applications (D 467 - 37 T):[7]

The following changes in permissible variations in dimensions and minor editorial changes are being made to correspond with revised standards of the National Electrical Manufacturers Association, since these particular features were originally based on the former N.E.M.A. standards:

Section 7.—Change Table IV from its present form to read as follows:

TABLE IV.—PERMISSIBLE VARIATIONS IN SIZE OF PIECES CUT FROM STANDARD SHEETS.

Nominal Thickness, in.	Permissible Variations in Length or Width, plus or minus, in.		
	6 in. and under	over 6 to 24 in.	24 in. and over
0.010 to 1/4, incl	0.010	0.015	1/32
17/64 to 1/2, incl	0.012	0.017	1/32
33/64 to 1, incl	0.015	0.020	1/32

Section 8.—In Table V, change the range in standard thickness for grade No. 1 from "$\frac{1}{64}$ to $\frac{3}{32}$ in., incl." to read "0.010 to $\frac{1}{4}$ in. incl."

Section 9.—Change Table VI from its present form to read as follows:

TABLE VI.—PERMISSIBLE VARIATIONS IN THICKNESS.

Nominal Thickness,[a] in.		Permissible Variations, plus or minus, in.	
Fraction of Inch	Decimal Equivalent	Grades Nos. 1, 2, 3, 4, and 6	Grade No. 5
0.010	0.010	0.002	.....
0.015	0.015	0.0025	.....
0.020	0.020	0.003	.....
0.025	0.025	0.0035	.....
1/32	0.031	0.0035	.....
3/64	0.047	0.0045	.....
1/16	0.0625	0.005	0.0075
3/32	0.094	0.007	0.009
1/8	0.125	0.008	0.010
5/32	0.156	0.009	0.011
3/16	0.1875	0.010	0.0125
7/32	0.219	0.011	0.014
1/4	0.250	0.012	0.015
5/16	0.3125	0.0145	0.0175
3/8	0.375	0.017	0.020
7/16	0.438	0.019	0.022
1/2	0.500	0.021	0.024
5/8	0.625	0.024	0.027
3/4	0.750	0.027	0.029
7/8	0.875	0.030	0.031
1	1.000	0.033	0.033

[a] On sheet of nominal thickness not listed in the above table, the permissible variations shall be the same as given for the next greater thickness.

Section 10.—Change last sentence in the first paragraph of footnote (*a*) of

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[5] 1939 Book of A.S.T.M. Standards, Part III, p. 656.

[6] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 230.

[7] 1939 Book of A.S.T.M. Standards, Part III, p. 702.

Table VII to read as follows by the addition of the italicized word and figure:

> This natural color shall be free from streaks or stains, and substantially uniform, except for grades Nos. *2 and* 3 which may be mottled in appearance.

Section 13.—In the first and second sentences change the requirement for marking the A.S.T.M. serial designation on sheets from "D 467 – 37 T" to read "D 467."

TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 39):[8]

A standard mold for producing test specimens for water absorption, dielectric strength, and power factor is being submitted for acceptance. While the present method does not include a test for power factor, this test is being studied and will probably be presented to the committee this year.

The following changes[9] are recommended as a tentative revision of these methods:

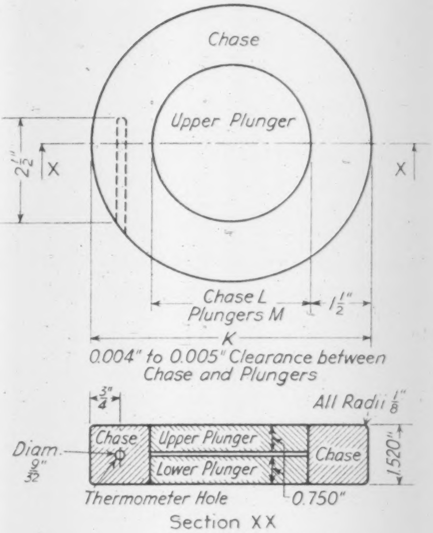
Section 20.—Add a new sentence at the end of this section to read as follows:

> The mold for making the test specimen is shown in Fig. 6 (the accompanying Fig. 1).

Section 30.—Change from its present form to read as follows:

> 30. The test specimen shall be molded in the form of a disk 2 in. (5.08 cm.) in diameter which should be $\frac{1}{8}$ in. (3.18 mm.) in thickness for hot-molded materials and $\frac{1}{4}$ in. (6.35 mm.) in thickness for cold-molded materials. The mold for making the test specimen is shown in Fig. 6 (the accompanying Fig. 1).
>
> NOTE.—To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimens will be permissible:

For hot-molded compounds....	±5 per cent
For cold-molded compounds....	±10 per cent



	Dimension K	Dimension L	Dimension M
Specimen for water absorption test ..	5 in.	2.032 in.	2.027 in.
Specimens for dielectric strength and power factor tests	7 in.	4.032 in.	4.027 in.

NOTE.—A top plate may be added, if the material to be handled in the mold is considered to require breathing.

FIG. 1.—Mold for Test Specimens for Water Absorption, Dielectric Strength, and Power Factor Tests.

Standard Methods of Testing Electrical Insulating Oils (D 117 – 40):[10]

The following tentative revisions in the dielectric strength test are recommended in order that this method will correspond with practices now being followed in various laboratories which represent improvements over the present procedure. The requirements for testing transformers and related equipment correspond with those for high-voltage testing apparatus prescribed in the Tentative Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 – 39 T). Two types of test are provided for: namely, an acceptance test, and a classification test for operating purposes. A clearer statement has been made of the accuracy to be obtained from the test.

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[8] 1939 Book of A.S.T.M. Standards, Part III, p. 244.

[9] This revision was further modified by Committee D-9 and accepted by Committee E-10 on Standards, see Editorial Note, p. 356.

[10] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 90.

Sections 20, 21, and 22.—Change these sections on the dielectric strength test from their present form to read as follows:

DIELECTRIC STRENGTH

20. *Apparatus: (a) Transformer.*—The desired test voltage may be most readily obtained by a step-up transformer energized from a variable low-voltage source. The transformer and its controlling equipment shall be of such size and design that, with the test specimen in circuit, the crest factor (ratio of maximum to mean effective) of the test voltage shall not differ by more than plus or minus 5 per cent from that of a sinusoidal wave over the upper half of the range of test voltage. The crest factor may be checked by means of a sphere-gap or peak-reading voltmeter in conjunction with an r.m.s. voltmeter. For test specimens of small capacitance, a testing transformer as small as 500 v.-amp. rating or a well designed potential transformer may be adequate. For specimens of high capacitance, a transformer of ample kilovolt-ampere rating must be used. Where the wave-form cannot be determined conveniently, a transformer having a rating of not less than 2 kva. shall be used for voltages not exceeding 50,000 v., and one having a rating of not less than 5 kva. for voltages exceeding 50,000 v. Tests shall be made at commercial power frequencies.

NOTE.—When a transformer is used at voltages lower than its full rating, the current drawn from the high voltage winding should not exceed the full-load full-voltage current rating.

(*b*) *Circuit Breaker.*—The test transformer circuit shall be protected by an automatic circuit-breaking device designed to open instantaneously on the current produced by breakdown of the test specimen. A prolonged flow of current at the time of breakdown causes carbonization of the liquid and pitting and heating of the electrodes, and thereby increases the work of electrode and test cup maintenance, and time of testing.

(*c*) *Voltage Control.*—The rate of voltage rise shall not, for short-time tests, vary more than plus or minus 25 per cent from the specified rate. Control of voltage may be secured in one of several ways:

(*1*) variable-ratio auto-transformer,
(*2*) resistance-potential divider,
(*3*) generator-field regulation, or
(*4*) induction regulator.

Preference should be given to equipment having an approximately straight-line voltage-time curve over the desired operating range. Motor drive, with variable speed control, should be preferred to manual drive because of the difficulty of maintaining a reasonably uniform rate of voltage rise with the latter.

(*d*) *Voltmeter.*—The voltage shall be measured by an approved method which gives root-mean-square values, preferably by means of:

(*1*) A voltmeter connected to the secondary of a separate potential transformer,
(*2*) An electro-static voltmeter in the high-voltage circuit, or
(*3*) A voltmeter connected to a well-designed tertiary coil in the test transformer.

A voltmeter connected to the low-voltage side of the testing transformer may be used only if the ratio of transformation does not change appreciably with load.

(*e*) *Rate of Rise of Voltage.*—The rate of voltage rise may be calculated from measurements of time required to raise the voltage between two prescribed values. When motor-driven regulating equipment is used, the speed-control rheostat may be calibrated in terms of voltage rise for any particular test transformer.

21. *Electrodes.*—The electrodes shall be polished metal disks 1 in. in diameter with square edges, mounted with axes horizontal and coincident.

22. *Test Cup.*—A special test cup with the electrodes rigidly mounted with their axes in a horizontal plane shall be used. The test cup should be of such design that no part of it is less than ½ in. from any part of the test faces of the electrodes, when the latter are in testing position. The total leakage and charging current of the test cup, when filled with a good grade of insulating oil, shall be less than 200 microamperes at 20 kv., 60 cycles. The test cup shall be made of a material of high dielectric strength, must be insoluble in, and unattacked by, any of the cleaning or test liquids, and nonabsorbent to moisture, and the cleaning and test liquids. Wood is not a suitable material for the test cup. The top of the cup should be about 1¼ in. (33 mm.) above the top of the electrodes.

23. *Adjustment and Care of Electrodes and Test Cup.*—(*a*) The electrodes and the test cup

shall be wiped clean with dry, calendered tissue paper or with a clean, dry chamois skin. The spacing of the electrodes of the test cup shall be checked with a standard round gage having a diameter of 0.100 in. or flat steel "go" and "no-go" gages having thicknesses of 0.0995 and 0.1005 in., respectively, and the electrodes locked in position. It is important to avoid touching the electrodes or the cleaned gage with the fingers or with portions of the tissue paper or chamois skin which have been in contact with the hands. The electrodes and cup shall then be rinsed with dry lead-free gasoline, Stoddard solvent, or dry, water-white kerosine until they are entirely clean. Care shall be taken not to touch the electrodes or the inside of the cup after cleaning, so as to avoid any possible contamination. After thoroughly cleaning the test cup, it shall be filled with a sample of the cleaning fluid and voltage applied with uniform increase at the rate of approximately 3 kv. per sec. (rms. value) until breakdown occurs. If the breakdown is not less than the established value of the oil being tested, the cup shall be considered in suitable condition for testing. If a lower value is obtained, the cup shall again be thoroughly cleaned and the test repeated. A cleaning fluid whose breakdown is not less than the established value of the oil being tested must be used.

(*b*) At the beginning of each day's testing, the electrodes shall be examined for pitting and carbon accumulation, and the spacing checked. The cup shall be thoroughly cleaned and tested as described in Paragraph (*a*). It shall then be flushed with a portion of the sample to be tested before it is filled for the test.

(*c*) If the test of a sample is below the breakdown value being used by the operator as a minimum satisfactory value, the cup shall be cleaned and prepared as described in Paragraph (*a*) before testing the next sample.

NOTE.—Evaporation of the cleaning fluid from the electrodes may chill them sufficiently to cause moisture to condense on their surfaces. For this reason, after the final rinsing with cleaning fluid, the cup must immediately be flushed with the liquid to be tested, following which the cup should be filled for the test.

24. *Preparation of Sample.*—The sample in the container shall be shaken vigorously so as to thoroughly mix the oil and allowed to stand for 15 min. to permit the air bubbles to rise to the surface. The container shall be inverted three times before filling the test cup. The cup shall be filled with the sample to a height of not less than ¾ in. (19 mm.) above the top of the electrodes and the sample allowed to stand in the cup for 3 min. prior to the first breakdown test and 1 min. prior to the application of voltage for each succeeding breakdown.

25. *Procedure:* (*a*) *Temperature of Test.*—The temperature of the sample when tested shall be the same as that of the room which should be between 20 and 30 C. (68 and 86 F.). Testing at lower temperatures is likely to give variable results which may be misleading.

(*b*) *Application of Voltage.*—Voltage shall be applied and increased at a uniform rate of 3 kv. per sec. from zero until breakdown occurs as indicated by a continuous discharge across the gap. (Occasional momentary discharges which do not result in a permanent arc may occur; these should be disregarded.)

(*c*) After each breakdown, a clean rod of smaller diameter than the gap shall be passed across the faces of the electrodes to remove any carbon particles that have adhered to the electrodes.

26. *Number of Tests:* (*a*) *Method A, Acceptance Tests.*—(This method must be used as a referee test between the seller and the purchaser.) Five breakdowns shall be made on each filling of the test cup after which the test cup shall be emptied and refilled with fresh oil from the original sample. The test shall be continued until the averaged values of each of at least three fillings do not differ from their mean more than 10 per cent. In any one filling, if an individual breakdown differs more than 25 per cent from the average for the filling, the results on that filling shall be discarded.

(*b*) *Method B, Classification Tests* (*Note*).—When it is desired for operating purposes to classify an oil with respect to an established breakdown value, tests on one filling may be sufficient. If the average of five breakdowns is more than 5 kv. higher than the established value or more than 5 kv. lower than the established value, the oil may be classified and no further tests shall be required. If the average of five breakdowns on the first filling lies within the range of plus or minus 5 kv. of the established value, two additional fillings shall be made and tested. The restrictions in method A relative to the deviation of the individual breakdowns per filling and deviation of the average of the fillings from the mean of the three averages shall also apply.

NOTE.—This procedure has been developed after a study of the breakdown data on 1145 samples. With the accumulation of further data, the range of plus or minus 5 kv. may be reduced. Many laboratories may use narrower limits after studying test results.

27. *Report.*—The report shall include the following:

(*a*) Procedure used, whether method A or B.

(*b*) The volts (r.m.s. value) at each breakdown, the average for each of the fillings, and the mean of the three fillings, if made. All results shall be reported to the nearest kilovolt,

(*c*) The breakdown stated in kilovolts for the 1.0-in. disk, 0.1-in. gap, and

(*d*) The approximate temperature of the oil at the time of the test.

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 39):[11]

Following the acceptance of the Proposed Tentative Method of Test for Water Absorption of Plastics (D 570 – 40 T) the following revisions in Method D 229 are recommended:

Section 19.—Change to read as follows by the addition of the italicized word and the omission of the words in brackets:

> 19. The water absorption tests are intended to determine the rate at which water is absorbed by the material when immersed *and* the total quantity absorbed at saturation [and the volatile content as received].

Sections 20 to 22.—Retain the heading for Method A and omit the present Sections 20 to 22, substituting for them the following new Section 20, and renumbering the subsequent sections accordingly:

> 20. The rate of water absorption of all sheet and plate insulating materials shall be determined in accordance with the Tentative Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570) of the American Society for Testing Materials.[12]

Section 24.—Renumber as Section 22 and change the first paragraph and Paragraph (*a*) from their present form to read as follows:

> 22. *Conditioning.*—(*a*) To determine the total water absorbed at saturation, three specimens shall be tested individually, weighed, and then conditioned as described in Paragraphs (*b*) and (*c*).
>
> (*b*) Materials whose water absorption value would be appreciably affected by temperature in the neighborhood of 110 C. shall be dried in an oven for 24 hr. at 50 ± 3 C., cooled in a desiccator, and immediately reweighed.
>
> (*c*) Specimens of materials such as phenolic laminated, vulcanized fiber, and other products whose water absorption value has been shown not to be appreciably affected by temperatures up to 110 C. shall be dried in an oven for 1 hr. at 105 to 110 C.

Renumber Paragraph (*b*) as Section 23 (*a*) and add a new heading to read "Procedure." Reletter Paragraph (*c*) as (*b*) and omit the second sentence of this paragraph which reads as follows:

> The percentage of water absorbed shall be calculated on the dry weight of the specimen.

Section 25.—Renumber as Section 24 and change the number of specimens referred to in the first sentence from "two" to read "three."

Section 26.—Renumber as Section 25 and change the heading from "Method C. Volatile Matter" to read "Volatile Matter."

Sections 27 and 28.—Renumber as Sections 26 and 27 and in the first sentence of each, change the number of specimens from "two" to read "three."

Section 41.—A summary of the round-robin data obtained on hardness tests indicates that the division between Rockwell M and R scales is necessary because of certain machines riding on the yoke when soft materials such as vulcanized fiber, cellulose acetate, and similar materials are being tested. Furthermore, the division cannot be made on the basis of the Rockwell values but must be made on the depth of penetration of the penetrator. Three new sentences at the end of Paragraph (*a*) are recommended for inclusion in this section, as follows:

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[11] 1939 Book of A.S.T.M. Standards, Part III, p. 265.

[12] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 292.

> Readings shall be taken on either the M or R scale. The M scale requires a $\frac{1}{4}$-in. ball penetrator, a minor load of 10 kg., a major load of 100 kg., and readings taken on the red scale. The R scale requires a $\frac{1}{2}$-in. ball penetrator, a minor load of 10 kg., a major load of 60 kg., and readings taken on the red scale.

Change Paragraph (*b*) from its present form to read as follows:

> (*b*) For all sheet materials other than vulcanized fiber, the choice of scales to be used shall be determined as follows: If readings with the M scale show a total indentation of less than 150 scale divisions on application of the major load, the readings shall be made on the M scale. If readings show a total indentation of 150 or more scale divisions, the tester shall be changed to the R scale combination. The total indentation shall be determined by the number of divisions the pointer passes through from the minor load position to the position when the major load is fully applied.

Omit the first two sentences of Paragraph (*c*) and substitute a new sentence to read as follows:

> For vulcanized fiber the R scale shall be used.

Section 42 (a).—Change from its present form to read as follows:

> (*a*) The Rockwell hardness number read on the red scale. The letter M or R shall precede the Rockwell hardness number depending on the combination which has been used in determining the hardness. The letter R shall precede all readings made on vulcanized fiber.

Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (D 348 - 39):[13]

The following revision involves a change in the water absorption test to conform with the new Proposed Tentative Method of Test for Water Absorption of Plastics.

Sections 10 to 12.—In place of the water absorption test described in these sections substitute the following, renumbering the subsequent sections accordingly:

> 10. The rate of water absorption shall be determined in accordance with the Tentative Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570) of the American Society for Testing Materials.[12]

Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (D 349 - 39):[14]

The following revision involves a change in the water absorption test to conform with the new Proposed Tentative Method of Test for Water Absorption of Plastics.

Sections 15 to 17.—In place of the water absorption test described in these sections substitute the following, renumbering the subsequent sections accordingly:

> 15. The rate of water absorption shall be determined in accordance with the Tentative Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570) of the American Society for Testing Materials.[12]

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Tentative Methods of Test for Conducting Paths in Electrical Slate (D 273 - 36 T):[15]

Although the use of slate for general switchboard applications has been largely superseded by the molded asbestos panelboard and other substitutes, it is felt that a need for this method still exists. Since this method has been published as tentative for four years without revision, it is recommended that it be adopted as standard.

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[13] 1939 Book of A.S.T.M. Standards, Part III, p. 257.

[14] *Ibid.*, p. 262.

[15] *Ibid.*, p. 776.

Tentative Methods of Testing Shellac Used for Electrical Insulation (D 411 - 37 T):[16]

These test methods have been in use for three years and have been found generally satisfactory. The test for polymerization has been found particularly satisfactory by the shellac industry and the method of test for flow has proved to be a great improvement over other tests existing in the industry prior to the adoption of this method. The test requires relatively simple apparatus and lends itself to much greater precision than has existed heretofore. These methods are accordingly recommended for adoption as standard.

ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

Standard Methods of Testing Electrical Insulating Oils (D 117 - 36):[17]

The committee recommends that the tentative revision of these methods published in June, 1935,[18] which provides for the inclusion of a reference to the Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (D 155 - 39 T) for determining the color of insulating oils, be referred to letter ballot of the Society for adoption as standard.

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[16] 1939 Book of A.S.T.M. Standards, Part III, p. 690.

[17] 1939 Book of A.S.T.M. Standards, Part III, p. 276.

[18] *Ibid.*, p. 1140.

REPORT OF SECTIONAL COMMITTEE
ON
ELECTRICAL INSULATING MATERIALS
ASA PROJECT: C59

Sectional Committee C59 on Electrical Insulating Materials was organized in 1934[1] under the procedure of the American Standards Association with the Society as sole sponsor. The scope of the committee covers "specifications and methods of test for electrical insulating materials."

Since its organization the committee has in general held at least one meeting, and sometimes two meetings, a year, the last meeting having been held in Washington, D. C., on February 27, 1940.

At the Washington meeting the present officers of the committee were reelected for the ensuing term of two years.

The committee has been interested in a revision that has been under consideration in the A.I.E.E. Standard No. 1 covering General Principles Upon Which Temperature Limits are Based in the Rating of Electrical Machinery and Apparatus. A special committee, appointed to study the standard with the idea of offering any suggestions for changes while the standard is in draft form, has reported that it feels the standard meets the need for an authoritative statement of the general principles involved in basing temperature limits for rating of electrical apparatus, and that it would be well for the revision to be used for a time as an A.I.E.E. standard before being submitted to the American Standards Association for approval as American standard.

Committee C59 has followed the activities of Committee No. 10 on Insulating Oils (of the International Electrotechnical Commission) through the technical adviser to the U. S. National Committee, E. A. Snyder. Cooperative work is under way with Dr. Bruckman of Holland, who is chairman of a special committee which is trying to develop a universal sludging test.

The scope of the I.E.C. Committee No. 15 has been enlarged from shellac to cover insulating materials in general, and the title of the committee has been changed to Insulating Materials. W. H. Gardner has been appointed technical adviser to succeed L. J. Cavanaugh, resigned. A draft British Standard Specification for Lac of the British Standards Institution has received very careful review by A.S.T.M. Committees D-1 on Paint and D-9 on Electrical Insulating Materials, and comments on the standard have been transmitted officially to the I.E.C.

Since the organization of Sectional Committee C59 in 1934, the following A.S.T.M. standards have been submitted to the American Standards Association on the recommendation of the sectional committee and have been approved as American standard:

Standard Methods of:

Testing Molded Materials Used for Electrical Insulation (A.S.T.M. D 48 – 39; ASA C59.1—1940),

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[1] See ASTM BULLETIN, No. 67, March, 1934, p. 7.

Testing Electrical Insulating Oils (D 117 – 36; C59.2—1937),
Test for Insulation Resistance of Electrical Insulating Materials (D 257 – 38; C59.3—1939),

Standard Specifications for:

Rubber Matting for Use Around Electrical Apparatus or Circuits not Exceeding 3000 Volts to Ground (D 178 – 24; C59.4—1935), and
Rubber Insulating Tape (D 119 – 38; C59.6—1939).

Approval was also given by the committee for the submittal to the ASA of the A.S.T.M. Standard Specifications for Friction Tape for General Use for Electrical Purposes (D 69 – 38) but this recommendation was withheld by the sponsor pending a further study on the part of A.S.T.M. Committee D-11 on Rubber Products of certain objections raised by the railroad interests.

The following A.S.T.M. standards are now being reviewed by special committees of the sectional committee looking toward their submittal to the ASA for approval as American standard:

Standard Methods of:

Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 39),
Test for Impact Resistance of Electrical Insulating Materials (D 256 – 38), and
Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 – 38).

A proposal that Sectional Committee C59 should serve as a repository for all standards of an authoritative nature in the field of electrical insulation is being followed, and a special committee under the chairmanship of G. E. Landt has been appointed to secure copies of the standards of the various organizations, including trade associations, that might be considered at all authoritative, with the idea of making them available to the sectional committee. When the standards have been collected the committee will give consideration to reviewing them looking toward their submittal to the ASA for approval as American standard.

Respectfully submitted on behalf of the sectional committee,

H. L. CURTIS,
Chairman.

R. E. HESS,
Secretary.

REPORT OF COMMITTEE D-11

ON

RUBBER PRODUCTS

Although Committee D-11 on Rubber Products, as a whole, has met only once since the 1939 annual meeting of the Society, its work has been advanced substantially by numerous subcommittee meetings and by correspondence. The committee meeting was held in Detroit, Mich., on March 7, 1940, in connection with the spring group meetings of A.S.T.M. committees.

The committee wishes to record its sorrow in the loss by death of C. R. Boggs. Mr. Boggs had been a member of the committee almost from its inception and, at all times, gave freely of his time and unusual talents for the advancement of the interests and work of the committee. He frequently served in official capacities, the most recent being as vice-chairman and acting chairman of Committee D-11. It is with a deep sense of its own severe loss that the committee extends its sympathy to Mr. Bogg's family and associates.

There have been two changes in subcommittee chairmen due to resignations, L. V. Cooper replacing J. J. Allen as chairman of Subcommittee XVII, and W. F. Tuley replacing E. B. Curtis as chairman of Subcommittee XVIII.

Subsequent to the 1939 annual meeting, Committee D-11 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of Testing Sponge Rubber Products (D 552 – 39 T),
New Tentative Methods of Test for Viscosity and Total Solids Content of Rubber Cements (D 553 – 39 T), and
Proposed Revision of Tentative Methods of Testing Hard Rubber Products (D 530 – 39 T).

These recommendations were accepted[1] by Committee E-10 on September 5, 1939, and appear in the 1939 Book of A.S.T.M. Standards.[2] All three of these methods are applicable to fields where practically no standardization exists at present.

The new Methods of Testing Sponge Rubber Products were referred to in the 1939 annual report.[3] Sponge rubber has, in recent years, been assuming increasing importance in industrial application for a variety of uses. Several different types of sponge are recognized which may be classified, first, as hard and soft, following the usual practice in distinguishing between hard rubber and ordinary soft rubber. They may be further classified, according to method of manufacture, into chemically blown sponge, expanded cell-type sponge, and foamed latex. These classes all have somewhat different properties and none of them can be tested by the methods used for the ordinary grades of rubber without modification. Prior to the formation of Subcommittee XXII, there had been practically no standardization in the field of sponge rubber. This subcommittee started work very actively immediately after its formation in June, 1938, undertaking as its first project, the standardization of test procedures. After surveying the field, certain tests were found to be of particular importance and capable of reduction to standard practice. The new methods accepted by the Society represent the results accomplished thus far. The subcommittee is entitled to great commendation for the diligence and speed with which it has advanced standardization in this field.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-11 reported the following results of the letter ballot vote of a total of 60 ballots returned from a committee membership of 105: D 552 - 39 T and D 553 - 39 T, affirmative 42, negative 0, ballots marked "not voting" 18; D 530 - 39 T, affirmative 44, negative 0, ballots marked "not voting" 16.

[2] 1939 Book of A.S.T.M. Standards, Part III, pp. 931, 928, 939.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 475 (1939).

A situation similar to that concerning sponge rubber has existed in the field of rubber cements. In the past there has been little standardization but great need for reliable methods of determining the quality of products offered for sale. Total solid content and viscosity are two properties which are very important. As mentioned in the 1939 report,[4] Subcommittee XXI had been carrying out extensive work in this field and the methods accepted for publication as tentative are the first part of this standardization project.

The Methods of Testing Hard Rubber Products, which were accepted for publication as tentative at the 1939 annual meeting and subsequently revised through Committee E-10, may likewise be considered the first effective standardization effort in connection with the quality control of hard rubber. There had been previous efforts along these lines but little was accomplished in the way of tangible results. Subcommittee XXIII has been working diligently since its organization in March, 1938, and it is especially noteworthy that this subcommittee has been able to bring into existence and agree upon methods of test in so short a time after all previous attempts failed.

The Standard Specifications for Rubber Insulating Tape (D 119 – 38) have been approved by the American Standards Association as American standard. The date of approval is July 27, 1939, and the A.S.A. number assigned to the standard is C59.6 – 1939.

Committee D-11, at its March meeting, decided to extend its scope to include rubber-like products. This change was approved on April 9, 1940, by the Executive Committee of the Society, and the revised scope now reads as follows:

> *Scope.*—The formulation of specifications and methods of test for products made from rubber or rubber-like materials, including research investigations concerning the properties of these materials.

The constantly increasing interest in the activities of the committee is evidenced by the number of new members who have been added during the year. The membership of the committee now totals 109, of whom 48 are classified as producers, 36 as consumers, and 25 as general interest members.

I. NEW TENTATIVE STANDARDS

Tentative Methods of Testing Automotive Hydraulic Brake Hose.[5]—These methods have been developed by Technical Committee A on Automotive Rubber and approved by Subcommittee I on Mechanical Rubber Hose in response to the urgent request of automotive engineers and state highway departments for suitable standards for the control of the hydraulic brake hose used on licensed vehicles on the highways. With the current widespread use of hydraulic brakes, inferior or defective hose introduces a very serious safety hazard. Heretofore, there has been no accepted standard for this material and many cases are on record where hose entirely unsuited to the service has been employed. Some state legislatures contemplate action in this connection, and the establishment of suitable standards is essential. In undertaking to develop these standards, Technical Committee A divided its program into two parts, one covering the preparation of standard purchase specifications giving the necessary quality requirements, and the other covering the development of standard test methods for evaluating the quality requirements. It was further decided to offer the specifications to the Society of Automotive Engineers for inclusion among its standards, and to submit the methods of test to the A.S.T.M. with the recommendation that they be accepted for publication as tentative.[5]

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 474 (1939).

[5] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 359.

Tentative Methods of Test for Compression-Deflection Characteristics of Vulcanized Rubber.[6]—There has long been a need for standard methods of compression testing of rubber although compression tests were the first to be applied in the case of vulcanized rubber. Progress has been much more rapid in tension testing than in compression testing. During recent years, service applications of rubber in compression for supporting loads and absorbing vibration have increased so rapidly that action in filling this need has become imperative. Also, since the compression properties of the material are so closely related to hardness, it has been thought that the load-deflection characteristics might be used in place of hardness figures for classification of different rubber compounds. This seems especially desirable because the properties so defined are often more directly related to the service requirements of a rubber article, and also because the methods more commonly used for determining the hardness of rubber have not proven entirely satisfactory. Therefore, Technical Committee A on Automotive Rubber, in collaboration with Subcommittee XVII, undertook to prepare test methods and to investigate their possibilities. The methods that Committee D-11 is recommending for publication as tentative[6] are the result of this work. Their utility and reproducibility have been demonstrated by an extensive cooperative test program on rubber compounds of both 40 and 60 Shore durometer hardness, tested independently in eight different cooperating laboratories.

Tentative Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method at 80 C.—The oxygen pressure method of accelerated aging was first issued as tentative in 1935 (A.S.T.M. Methods: D 428) although this test had been in common use at that time for about ten years. During all of the period up to the present, the conditions under which the rubber was exposed to the action of oxygen have been specified as 300-lb. oxygen pressure and 70 C. This same period has marked the development and introduction into common use of antioxidants which are incorporated in rubber compounds to improve their resistance to oxidation. As a result, the exposure periods of the test, during which the deterioration agencies act on the rubber, have steadily become increasingly longer. It has been felt for some time that measures should be taken to make the method more severe and decrease the time required for conducting the test. Unfortunately, this problem is not simple because the oxidation reaction is quite complex and the exact mechanism of the deterioration of rubber is none too well understood. It was known that higher temperatures would accomplish the results desired but considerable evidence was advanced showing that, at temperatures above 90 C., the nature of the deterioration is different and the proportional rate of change deviates from that expected. Also, the accumulated experience of 15 years, which has thoroughly established the utility and reliability of the original test conditions, cannot lightly be discarded in favor of new ones without more experience than can be gained quickly in cooperative testing. Considerable test data have been considered by the committee involving exposure temperatures of 80 and 85 C. as well as the established 70 C. The committee is now proposing to adopt as standard, as recommended later in this report, the original oxygen aging procedure described in Methods D 428 but, in order to meet the need for a shorter test, it is recommending for publication as a tentative revision[7] of the newly adopted standard a procedure using 80 C. in place of 70 C., with the thought that in due time, after sufficient experience has been gained by actual trial, the test at 80 C. may replace the former one at 70 C.

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[6] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 338.

Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation.[8]—These specifications, which are recommended for publication as tentative, have been prepared by Subcommittee V after considerable investigation extending over several years. They are intended to cover a type of insulation compound, designed specifically to resist the action of ozone which is frequently encountered in electrical circuits, particularly carrying the higher voltages. The present insulated wire specifications are not considered to be adequate in this respect.

II. REVISIONS OF TENTATIVE STANDARDS

The committee is proposing revisions of several tentative specifications and methods of test, as indicated below, and recommends that the tentative standards as revised be continued as tentative.

Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 39 T):[9]

Section 2.—At the end of this section add two new sentences to read as follows:

> Metallic coatings other than tin may be used if approved by the purchaser. Coatings conducive to galvanic action at splices, such as zinc, are not recommended.

Section 29.—At the end of this section add a sentence to read as follows:

> Other types of tape may be used if approved by the purchaser.

Table V.—Change Note 5 to read as follows by the addition of the italicized words and the omission of those in brackets:

> NOTE 5.—Above 5000 v. alternating-current, [special high-voltage rubber compounds are preferred according to manufacturer's recommendations] *ozone-resistant insulation is recommended as prescribed in the Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation (A.S.T.M. Designation: D 574) of the American Society for Testing Materials.*[10]

Change the minimum conductor sizes for various voltage ratings in Table V to agree with those prescribed in the new Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation, as follows:

> For 2001 to 3000 v. and for 3001 to 4000 v., change the minimum conductor size from "No. 14 to No. 8" to read "No. 10 to No. 8."
>
> For 4001 to 5000 v., change the minimum conductor size from "No. 14 to No. 8" to read "No. 8."

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[7] This revision was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 570.

[8] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 396.

[9] 1939 Book of A.S.T.M. Standards, Part III, p. 886.

[10] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 396.

For 5001 to 6000 v., grounded and ungrounded, change the minimum conductor size from "No. 14 to No. 0000" to read "No. 8 to No. 0000."

Tentative Specifications for Insulated Wire and Cable: Performance Rubber Compound (D 353 – 39 T),[11] *and for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 – 39 T)*:[12,13]

Table II.—Make the same changes in this table and in Note 5 as are recommended above in Table V of Specifications D 27 – 39 T.

Tentative Methods of Testing Flat Rubber Belting (D 378 – 39 T):[14]

Section 4 (d).—At the end of this section add two new sentences to read as follows:

> When determining the cover thickness of a belt it is essential to have the specimen lie flat on the platform of the micrometer and the projecting ends must be properly supported to prevent tilting. The test specimen may consist of a small section cut from the belt.

Section 8 (a).—At the end of this section add a new sentence to provide for the direction in which the tension specimen shall be taken, to read as follows:

> The test specimens shall be stamped from the covers in a transverse direction, at right angles to the axis of the belt.

Oil Immersion Test.—Add as new Sections 10 to 16, renumbering the subsequent sections accordingly, the oil immersion test appended hereto.[15]

Section 15 (a).—Change the next to the last sentence from its present form: namely,

> If the specimen breaks in the jaws the results shall be discarded and the test shall be repeated.

to read as follows:

> Test specimens that break in the jaws or that, upon examination, are found to be imperfect shall be discarded, and retests shall be made unless the results obtained in the first test exceed minimum values that may be required by detailed specifications.

Tentative Methods of Testing Rubber Hose (D 380 – 39 T):[16]

The revisions given below are recommended so that the methods will conform to best current practice.

Section 16.—Change the next to the last sentence to read as follows by the addition of the italicized words and figures:

> The petcock shall then be closed and the pressure shall be applied at a uniform rate of increase of approximately 1000 psi. per min., *except for hose with bursting strength specified above 2000 psi., in which case the pressure shall be applied at a rate of 10,000 psi. per min. or as rapidly as pump capacity will permit.*

Following this section add a note to read as follows:

> NOTE.—The specified rate of 1000 psi. per min. requires too much time for small-diameter high-strength hose, such as hydraulic brake hose, wire reinforced grease gun hose, etc. With $\frac{1}{8}$ in. hydraulic brake hose, pressure increases at rates as high as 40,000 psi. per min. are satisfactory.

Section 17.—Change the heading of this section from "Bursting Test" to read "Bursting Tests"; designate Paragraph (*a*) as "Straight Bursting Test," Paragraph (*b*) as "Curved Bursting Test," and add a new Paragraph (*c*) covering "Hold Test," to read as follows:

> (*c*) *Hold Test.*—When a hold test is specified instead of a bursting test, pressure shall be applied as described in Section 16 until the required pressure is reached. The specimen shall withstand the specified pressure for a

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[11] 1939 Book of A.S.T.M. Standards, Part III, p. 908.

[12] *Ibid.*, p. 902.

[13] A further revision in these specifications was submitted subsequent to the annual meeting, see Editorial Note, p. 380.

[14] 1939 Book of A.S.T.M. Standards, Part III, p. 873.

[15] This revision was accepted by the Society and has been incorporated in the methods which appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 365.

[16] 1939 Book of A.S.T.M. Standards, Part III, p. 863.

10-min. period without bursting, leaking, or developing other defects. Specimens which have been subjected to this test shall not be considered fit for service.

Section 24 (e).—In the sixth sentence change the specified drying time from "24 hr." to read "4 hr."

Tentative Methods of Test for Compression Set of Vulcanized Rubber (D 395 - 37 T):[17, 18]

A less expensive compression device using a different method of applying the spring load from that now specified in Method A has been developed and found to be equally satisfactory. Also, it is considered essential for accurate results, to control the temperature of the compression device at the beginning of the test. The following revisions are therefore recommended:

Sections 7 and 8.—Combine these sections into a new Section 7 to read as follows, renumbering the subsequent sections accordingly:

7. *Compression Device*.—The compression device shall consist of a loading spring and two parallel compression plates assembled by means of a frame or threaded bolts in such a manner that the device shall be portable and self-contained after the load has been applied and that the parallelism of the plates shall be maintained. The load may be applied in either of the following ways:

(*a*) *Calibrated Spring Loading*.—In this case the required load shall be applied by a screw mechanism for compressing a calibrated spring the proper amount as measured to the nearest hundredth of an inch. The spring shall be of properly heat-treated spring steel with ends ground square and perpendicular to the longitudinal axis of the spring. A suitable compression device is shown in Fig. 1.[a]

The spring shall conform to the following requirements: The spring shall be calibrated at room temperature (24 ± 5 C. (75 ± 9 F.)) by applying successive increments of load of 50 ± 0.5 lb. and measuring the corresponding deflection in hundredths of an inch. The curve obtained by plotting the loads against the corresponding deflections shall have a slope of 4.0 ± 0.2 at 400-lb. load. The slope is obtained by dividing the difference between the two loads expressed in pounds by the difference between the corresponding deflections expressed in hundredths of an inch.

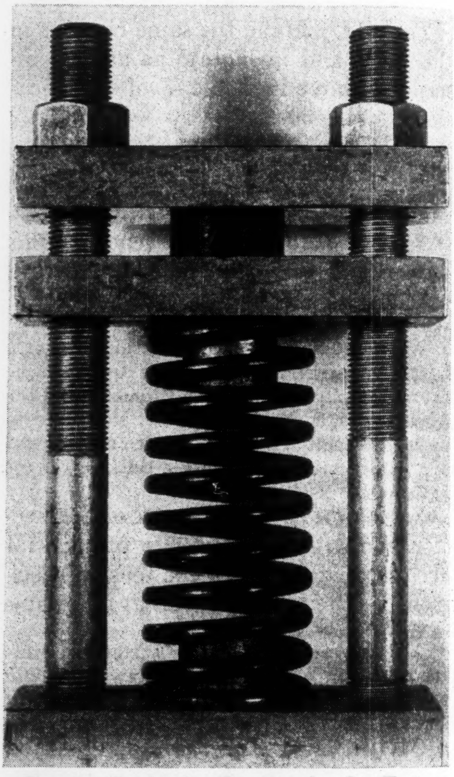

FIG. 1.—Device for Compression Set Test, External Loading.

Note: *Example*.—A spring which deflects 1.00 in. at 400 lb. and 0.75 in. at 300 lb. gives a slope of 4.0.

The original dimensions of the spring shall not change due to fatigue by more than 0.01 in. after it has been mounted in the compression device, compressed under a 400-lb. load and heated in the oven for 1 week at 70 C. (158 F.). In ordinary use, a weekly check of the dimensions shall show no greater change than this over a period of 1 yr.

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[17] 1939 Book of A.S.T.M. Standards, Part III, p. 839.

[18] A further revision in these methods was submitted subsequent to the annual meeting, see Editorial Note, p. 380.

[a] This refers to Fig. 1 in the present method, see 1939 Book of A.S.T.M. Standards, Part III ,p. 841.

The minimum load required to close the spring solid shall be 530 lb.

(*b*) *External Loading.*—In this case the required load shall be applied to the compression plates and spring by external means after the test specimen is mounted in the apparatus. Either a calibrated compression machine or dead weights may be used for load application. Provision shall be made by the use of bolts and nuts or other devices to prevent the specimen and spring from losing their initial deflections when the external load is removed. The spring shall have essentially the same characteristics as described in Paragraph (*a*), but calibration is not required. A suitable compression device is shown in Fig. 2 (the accompanying Fig. 1).

Section 9.—Change Paragraph (*b*) to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

(*b*) Application of Load.—The test specimen shall be assembled in the compression device using extreme care to place it exactly in the center between the plates so that tilting may be avoided. [The spring shall be compressed] *If the calibrated spring device is used, the load shall then be applied* by tightening the screw until the deflection as read from the scale is equivalent to that shown on the calibration curve for the spring as corresponding to a load [on the sample] of 400 lb. *With the external loading device, a 400-lb. load shall be applied to the assembly in the compression machine or by dead weight, but in the latter case care must be taken to apply the weight gradually without shock. The nuts and bolts shall then be tightened just sufficiently to hold the initial deflections of the specimen and spring. It is imperative that no additional load be applied in tightening the bolts.*

Add three new sentences at the beginning of Paragraph (*c*) to read as follows:

Before inserting the test specimens in the compression device, the compression device shall be preheated sufficiently long to insure that the entire assembly is at the testing temperature. A minimum amount of time shall be consumed in changing or in inserting test specimens. Should it be necessary to keep the device out of the oven longer than 20 min., it is suggested that the device after loading shall be immersed in water at 158 F. for a period of 20 min. before being replaced in the air oven.

Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 – 37 T):[19,18]

Sections 7 and 8 (*a*).—It is recommended that the reference to 95 per cent ethyl alcohol be changed to read "SDA-2B alcohol." The reason for this is that very few laboratories have available 95 per cent ethyl alcohol whereas permits for SDA-2B alcohol, which is 95 per cent ethyl alcohol containing 0.5 per cent benzene, may be obtained without difficulty.

III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the tentative methods listed below be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated.

Tentative Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 – 37 T):[20]

These methods as originally issued in 1934 included six procedures, each using different abrasion machines which were then commonly employed and also complete instructions for the preparation of comparison standards. Since that time, considerable standardization has taken place and the committee is now of the opinion that three of these methods may be eliminated. The remaining three employ machines which are commercially available and will probably remain in use indefinitely. In general, preference seems to be given in most laboratories to the method originally published as Method B using the E. I. du Pont de Nemours and Co. abrader. The committee has arranged with the V. L. Smithers Laboratories to make available certified reference standard specimens

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[19] 1939 Book of A.S.T.M. Standards, Part III, p. 835.

[20] *Ibid.*, p. 824.

for this method. It is now recommended that Tentative Methods D 394 – 37 T be revised by eliminating Method C using the B. F. Goodrich Co. abrader, Method D using the Kelly-Springfield abrader, and Method E using New Jersey Zinc Co. abrader. In the revised methods, the procedures employing the E. I. du Pont de Nemours and Co. abrader will be designated Method A, the National Bureau of Standards abrader as Method B, and the United States Rubber Co. abrader as Method C.

Tentative Methods of Test for Accelerated Aging of Vulcanized Rubber (D 428 – 36 T):[21]

These methods as originally issued in 1935 included procedures for two types of test which have been in common use for a number of years. The older method, called the air-oven aging test, involved the exposure of rubber samples in an oven to the action of atmospheric air at 70 C. for extended periods and measurement of the extent of the deterioration. In the other method, called the oxygen-pressure-chamber aging test, the samples were exposed to the action of pure oxygen at 300-lb. pressure and 70 C. temperature instead of to atmospheric air. Both of these methods are satisfactory as standardized and the committee recommends their adoption as standard. In adopting the methods, however, a rearrangement is recommended which is to consist of separating the two types of tests into separate standards to be designated, respectively, "Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method" and "Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method."[22] In separating these methods, no change is being made in substance or procedure. The change seems advisable because it is now proposed, as mentioned earlier in this report, to present for publication as a tentative revision of the newly adopted standard an oxygen pressure method in which the exposure of the specimens is made at a higher temperature.

Tentative Methods of Test for Flexing of Rubber Products (D 430 – 35 T):[21]

The committee recommends the adoption as standard of these tentative methods with certain minor editorial and technical revisions. Subcommittee VIII on Nomenclature has ruled that the term "flexing" refers to bending and does not apply to either one or several cycles of extension or retraction as used in these methods. Therefore, the committee proposes to change the title of the methods to read "Standard Methods of Dynamic Testing for Ply Separation and Cracking of Rubber Products," and also to change the text wherever the word "flexing" has been misused according to the above definition and to substitute appropriately the terms "dynamic fatigue," "fatigue cracking," or "strain."

In addition, the following revisions are recommended:

Section 19.—Between the third and fourth sentences add three new sentences to read as follows:

> The specimens may be mounted on the machine most conveniently by holding them properly spaced in parallel positions in a special rack. The distance between the outer edges of the side bars of the rack shall be equal to the space between the jaws of the testing machine when positioned for holding the specimens without tension. The specimens can be mounted on the testing machine by bringing the jaws into contact with the mounting rack and tightening the clamps on the projecting ends of the specimens.

Section 26.—Correct an error in the seventh sentence by changing the belt tension from "33 lb." to read "17 lb."

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[21] 1939 Book of A.S.T.M. Standards, Part III, p. 815, 844.

[22] These methods were adopted as standard by the Society, and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 102 and 107, respectively.

The committee also recommends that the tentative standards listed below be approved for reference to letter ballot of the Society for adoption as standard without revision. They are believed to be satisfactory, having had a tentative status for three years, during which time either none or very minor criticisms were received.

Tentative Method of Air Pressure Heat Test of Vulcanized Rubber (D 454 – 37 T),[23] and

Tentative Specifications for Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground (D 120 – 37 T).[24]

IV. TENTATIVE STANDARDS CONTINUED WITHOUT REVISION

The committee recommends that all tentative standards under its jurisdiction not mentioned in this report be continued as tentative without revision. The Tentative Method of Test for Resistance to Light Checking and Cracking of Rubber Compounds (D 518 – 38 T) has had a tentative status for two years without revision. The committee has been carrying on extensive work in connection with accelerated light tests of rubber, as mentioned later in this report, and it is desirable that this method be continued in its present form until this work reaches a conclusion.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 109 members; 66 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

The subcommittees of Committee D-11 have been exceedingly active in their respective fields during the year. Because of the wide variety and extent of these activities, only a few of the most important may be discussed here.

Technical Committee A on Automotive Rubber (L. A. Danse, chairman).—Organization of this technical committee was mentioned in the 1939 report.[25] The personnel of the committee, designated jointly by Committee D-11 and the Society of Automotive Engineers, consists of 24 members and, as

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Testing Automotive Hydraulic Brake Hose	32	0	34
Test for Compression-Deflection Characteristics of Vulcanized Rubber	44	0	22
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method at 80 C.	49	2	15
Spec. for Insulated Wire and Cable: Ozone-Resistant Insulation	32	3	31
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 39 T)	33	0	33
Spec. for Insulated Wire and Cable: Performance Rubber Compound (D 353 - 39 T)	33	0	33
Spec. for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 - 39 T).	35	0	
Testing Flat Rubber Belting (D 378 - 39 T)	33	0	33
Testing Rubber Hose (D 380 - 39 T)	35	1	30
Test for Compression Set of Vulcanized Rubber (D 395 - 37 T)	42	0	24
Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 - 37 T)	44	0	22
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Abrasion Resistance of Rubber Compounds (D 394 - 37 T), as revised	45	0	21
Test for Accelerated Aging of Vulcanized Rubber (D 428 - 36 T)	54	0	12
Test for Flexing of Rubber Products (D 430 - 35 T), as revised	42	0	24
Air Pressure Heat Test of Vulcanized Rubber (D 454 - 37 T)	28	1	37
Spec. for Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground (D 120 - 37 T)	53	0	13

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[23] 1939 Book of A.S.T.M. Standards, Part III, p. 820.

[24] *Ibid.*, p. 880.

[25] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 471 (1939).

finally constituted, includes 10 representatives of producers of automotive rubber products, 11 consumer representatives from the automotive and aircraft industries, and 3 members-at-large representing the U. S. Army Ordnance Department, the Army Air Corps, and the Navy Bureau of Construction and Repair.

The first meeting of the committee was held in Detroit, Mich., on June 8, 1939, at which time a work program on automotive standards was outlined and decision made to start with the material used for rubber motor mountings. It was at once apparent that this work naturally divided into two phases, the first dealing with the classification of the various types of material used according to physical properties, with a view toward ultimately establishing standard specifications based on these properties, and the second covering the development and standardization of suitable tests for determining the required physical properties. It was decided by the committee that the definition of rubber compounds for automotive service by standardization of composition and chemical requirements is impracticable for the most part and that the work should be confined to physical and mechanical properties. The technical committee has held seven additional meetings during the year with many other projects added from time to time. The closest possible cooperation has existed between the various interests represented and the record of accomplishment in so short a period is indeed imposing.

During the early meetings, three sections of the technical committee were established: I on Classification of Rubber Motor Mountings, II on Classification of Rubber Bumper Parts, and III on Tests. It was soon found that better progress might be made by having the product sections cover both classification and tests for each product, whereupon Section III, which had by that time completed a survey of existing test methods used in current automotive specifications, was then reconstituted as a section on Development of Standards for Hydraulic Brake Hose.

The Section on Motor Mountings found that it is customary in the automotive industry to class rubber for motor mountings by different grades of hardness, usually defined in terms of the reading by the Shore durometer. Much dissatisfaction exists with this method, and the A.S.T.M. hardness instrument has not proven to be a satisfactory substitute because of lack of convenience and adaptability in use for inspection purposes. The section decided that better results could be obtained by classifying the rubber according to its compression-deflection characteristics, although the desirability of a satisfactory indentation hardness test which would employ a small hand instrument having both convenience and accuracy was still fully appreciated. Work was, therefore, directed toward the accomplishment of both of these ends, namely, the preparation of a method of compression-deflection testing and the securing of a suitable hand hardness tester. In developing the compression-deflection method, extensive technical studies were made with eight laboratories cooperating and the result is presented elsewhere in this report. Work is continuing on this method but is now directed toward the standardization of apparatus in which the committee has the cooperation of four manufacturers of testing equipment. A great deal of effort has been expended in connection with the problem of obtaining a suitable hardness tester. Four newly designed instruments have been submitted to the committee by the manufacturers and have been investigated by a number of laboratories. This work, however, has not yet reached a satisfactory conclusion. With the completion of the compression-deflection method of testing, the section prepared a classification of motor mounting stocks, specifying the properties of hardness, compression-deflection, maximum compression set, and minimum adhesion to metal on five different grades. It was planned to recommend this as an S.A.E. standard but the section withheld such action when it was found that further work was needed on the method for determining compression set and additional checking seemed desirable on the load-deflection tests.

The Section on Bumper Parts has also carried out very extensive investigations and test programs. Bumper materials were originally classified in three grades according to hardness. This section has reached agreement with the Section on Motor Mountings relative to the use of hardness and compression-deflection methods. The question of impact has presented a serious problem to this section because bumpers may fail either by rupture, as the result of sudden severe shock, or by heat failure and disintegration as the result of repeated deflection with rapid frequency. Suitable tests to evaluate these characteristics have not yet been agreed upon and the section is devoting very serious study to the problem in an effort to develop and standardize such tests.

Most of the work of Section III on Hydraulic Brake Hose has been already discussed earlier in this report in connection with the recommendation on the Tentative Methods of Test for Hydraulic Brake Hose. Since the completion of these methods, and the specifications which are being presented as an S.A.E. standard, all of which was accomplished with remarkable rapidity, a new project relating to automotive hose has been undertaken. The attention of the section at the January meeting was called to the fact that considerable difficulty is being encountered in automotive service from the coolants which come in contact with the hose used in radiators and car heating systems. Measures are urgently needed to eliminate this situation, either by assuring the use of properly resistant hose or by preventing the use of improper coolants. The first step to this end would be the development of suitable test methods, and the section is now actively at work on this problem.

In connection with the extensive work which has been done, the technical committee has frequently felt the need of a suitable means of obtaining fundamental information concerning properties of rubber and methods of test where such information is not now available. A proposal was made that the committee consider the establishment of a technical research foundation through which such information might be obtained. This proposal has been given serious study but no decision has as yet been reached because of the formidable difficulties which the proposal presents due to the magnitude of its scope and the problem of adequately financing such a program. The need for such cooperative research is very definitely recognized and it is hoped that further study may offer some means of overcoming the difficulties.

Subcommittee I on Mechanical Rubber Hose (E. G. Kimmich, chairman).—This subcommittee has been cooperating actively with Technical Committee A in its work on hydraulic brake hose. It has now had referred to it by the technical committee the problem, mentioned above, of automotive radiator and car heating system hose. A section has been organized and work has been started on this problem.

Subcommittee VI on Packings (William Staniar, chairman).—This subcommittee has had its name changed from "Packings, Gaskets, and Pump Valves" to "Packings." According to the definitions which it has adopted, a gasket is a special form of packing, and a valve disk is a special type of gasket. The term "packing", therefore, is the more general term.

The section, mentioned in the 1939 report,[26] that was appointed to survey the methods of test in current use, completed its work and prepared a digest of its activities, a summary of which has been given by the secretary of the subcommittee, Frederick Thorn, in the ASTM BULLETIN.[27]

The section that was organized to prepare standards for sheet packings considered a proposed draft which was based on the conventional tests, but after detailed study it felt that these tests were inadequate and that the work should be deferred pending a more comprehensive study of test methods, particularly of the performance type. A special section on Stress Decay in Packings has been organized and is already at work on the problem of development of better tests for packings.

While Subcommittee VI has not yet found itself in a position to recommend any new standards, a great amount of work has been done and a foundation is being laid upon which sound future progress may be based.

Subcommittee XI on Chemical Analysis of Rubber Products (S. Collier, chairman).—This subcommittee has continued its investigation of methods for the determination of small amounts of manganese and copper in rubber and has completed the additional cooperative studies mentioned in last year's report.[26] It is expected that definite recommendations may be forthcoming which will be presented to the Society through Committee E-10 on Standards subsequent to this annual meeting.[28]

The section that was organized to study and write revised procedures for the Standard Methods of Chemical Analysis of Rubber Products (D 297 - 39) has been actively at work. This section has cooperated with the Federal Specifications Executive Committee in revising the methods appearing in the Federal Specification for Rubber Goods (ZZ-R-601). As a result of this work, there are a number of changes in the Society's methods which should be made immediately to bring them in line with the Government standard. In addition, the section contemplates the inclusion of methods of ash analysis as well as several other changes and additions. In view of this situation, it seems probable that the committee will recommend in the near future that the present Standard Methods of Chemical Analysis of Rubber Products (D 297 - 39), be reverted to the tentative status until all of the contemplated changes can be incorporated in them.[28]

Subcommittee XIV on Abrasion Tests for Rubber Products (G. S. Haslam, chairman).—In addition to the recommendation that the Tentative Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 - 37 T) be adopted as standard, as mentioned earlier in this report, this subcommittee has prepared a proposed tentative method for determining the tear resistance of vulcanized rubber. This work was done at the request of Technical Committee A, many of whose members have expressed need for such a test. Unfortunately, the best available technical information seems to indicate that none of the present tear tests is reliable as a true measure of tear resistance under all service conditions. The subcommittee has been forced, therefore, to select the procedure which is in widest use and prepare this in standard form, though it recognizes that the method is not entirely satisfactory. This subcommittee has handled the assignment because abrasion has been defined as a combination of tearing and cutting. The subcommittee expects to present the new test method to the Society through Committee E-10 on Standards[28] in the near future.

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[26] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, pp. 472, 473 (1939).

[27] ASTM BULLETIN, No. 104, May, 1940, p. 17.

[28] See Editorial Note, p. 280.

Subcommittee XV on Life Tests for Rubber Products (J. H. Ingmanson, chairman).—In addition to the work which is represented by the recommendations mentioned earlier in this report dealing with accelerated aging tests, this subcommittee has given a great deal of attention during the year to methods of testing rubber compounds for resistance to light aging. Two years ago the subcommittee prepared a Tentative Method of Test for Resistance to Light Checking and Cracking of Rubber Compounds (D 518 – 38 T). This method is widely used but is not adequate because of the natural wide variations in quality and duration of sunlight in different geographical locations. Such tests are also necessarily extended over long periods and there is great need for an accelerated light test which can be carried out in the laboratory under controlled artificial light conditions. In attacking this problem it was felt that the first requisite would be a means whereby the intensity and quality of radiation of a light source selected as standard could be calibrated. A suitable method is now available and is based on the fact that oxalic acid in the presence of uranyl sulfate in aqueous solution is photochemically decomposed by light essentially at a rate in direct proportion to the intensity and total radiation of the light source to which the solution is exposed over a given wave-length range selected for the test. The calibration method, therefore, involves exposure of a standardized oxalic acid – uranyl sulfate solution in a quartz cell to the radiation to be evaluated, followed by a determination of the quantity of oxalic acid decomposed during a measured time period of exposure. This calibration method may also be adapted for measuring and integrating the intensity or total exposure from other light sources which are intermittent or variable, such as sunlight. The subcommittee has also prepared a proposed tentative method of testing rubber compounds for resistance to accelerated light aging using a standardized artificial light source in a specified, specially designed apparatus which includes the light source, suitable filters, a water spray, and a revolving hood with facilities for mounting specimens for exposure. It is expected that both the method for calibration of the light source and the method for testing rubber specimens under the artificial light will be presented to the Society through Committee E-10 on Standards this summer.[28]

Subcommittee XXIII on Hard Rubber (W. H. Juve, chairman).—Following the approval for publication in 1939 of the Tentative Methods of Testing Hard Rubber Products (D 530 – 39 T) the subcommittee felt that further work should be done to make certain that the best methods had been selected and that various laboratories testing hard rubber products could satisfactorily duplicate results by their use. An extensive cooperative testing program was, therefore, undertaken and is now in progress. Three typical qualities of hard rubber having known compositions have been prepared at central points and the necessary samples for complete chemical, physical, and electrical tests have been supplied to 16 cooperating laboratories. It is expected that valuable criticisms and suggestions for improvement may result from this program in addition to a substantial amount of data relative to the present methods.

As a new project, the subcommittee also has under consideration a standardization program on material used in manufacturing the asphalt type of battery container.

This report has been submitted to letter ballot of the committee which consists of 109 members; 66 members returned their ballots, of whom 53 have voted affirmatively and 0 negatively.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, O. M. Hayden.
Vice-Chairman, J. J. Allen.
Secretary, Arthur W. Carpenter.

The following members with the above officers were elected to constitute the advisory subcommittee: S. Collier, G. S. Haslam, M. E. McDonnell, and R. A. Schatzel.

Respectfully submitted on behalf of the committee,

O. M. HAYDEN,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-11 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Methods of:
- Test for Compression Set of Vulcanized Rubber (D 395 - 40 T),
- Tension Testing of Vulcanized Rubber (D 412 - 39 T),
- Testing Rubber Insulated Wire and Cable (D 470 - 39 T),
- Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 - 40 T),

Revision of Tentative Specifications for:
- Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 - 40 T),

Tentative Revisions of Standard Methods of:
- Sample Preparation for Physical Testing of Rubber Products (D 15 - 39),
- Chemical Analysis of Rubber Products (D 297 - 39), in the form of new Tentative Methods of Chemical Analysis of Rubber Products (D 297 - 40 T).

These recommendations were accepted by Committee E-10 on August 28, 1940, and the tentative standards in their revised form and the tentative revisions appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 332, 320, 410, 327, 390, 570, and 295.

Committee D-11 also submitted Proposed Tentative Methods of Test for Resistance to Accelerated Light Aging of Rubber Compounds, Calibrating a Light Source Used for Accelerating the Deterioration of Rubber, and Test for Tear Resistance of Vulcanized Rubber, but in view of the number of negative votes cast on these items, the methods were not approved by Committee E-10 for publication as tentative but instead the three proposed methods are published as information, as appended to this report on pages 381, 385, and 389.

On September 26, 1940, Committee E-10 accepted for publication a new Tentative Method of Test for Physical State of Cure of Vulcanized Rubber (T-50 Test), and approved the withdrawal of the Standard Methods of Chemical Analysis of Rubber Products (D 297 - 39). The new tentative method appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 341, bearing the designation D 599 - 40 T.

APPENDIX I

PROPOSED METHOD OF TEST FOR RESISTANCE TO ACCELERATED LIGHT AGING OF RUBBER COMPOUNDS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method is intended for use as a static accelerated test applicable for estimating the comparative resistance of soft vulcanized rubber compounds to deterioration when exposed to light having a frequency range approximating that of sunlight but having a greater intensity in the ultraviolet range than sunlight. It is believed that the range of wave lengths which are particularly instrumental in causing the cracking of rubber lie in the range of 295 to 335 mμ. This method is not intended to cover the testing of materials ordinarily classed as hard or semihard rubber.

Type of Test

2. This test consists of continuously or intermittently exposing rubber specimens held in a jig or holder under a specified strain to the effect of light having essentially the same wave lengths as are found in natural sunlight but with increased intensity in the ultraviolet range. The frequency of the light to which the rubber specimens are exposed is limited in the lower range by filtering through a Corex D filter which transmits from the region of 295 mμ. The primary criterion to be used in estimating resistance to light aging shall be the percentage decrease in tensile strength and in elongation at break. A supplementary criterion for estimating resistance to light aging shall be the observed extent of surface crazing and cracking.

Apparatus

3. The apparatus shall consist of a light source, filters, a suitable revolving hood, facilities for mounting specimens for exposure, a water spray, power equipment, and controls. The apparatus shall preferably conform to the requirements prescribed in the following Paragraphs (*a*) to (*h*), but equipment other than that described will be acceptable providing the range of light radiation is approximately the same as that produced in the apparatus described:

(*a*) The light source shall be of the flaming arc type. The lamp shall have a dual trim mechanism and shall be designed to operate on 230 v., 60 cycle alternating-current circuits. The arc shall operate normally at 60 amp. and 50 v. The lamp shall accommodate two pairs of carbons but shall burn between only one pair of carbons at a time. When one set of carbons burns back, the arc shall shift to the other pair without interruption. The carbons used shall be cored and copper coated. They shall be of such length that when operating at 60 amp. and 50 v. the two pairs shall operate for approximately 9 hr. The lamp shall be mounted on a suitable framework equipped with cables, pulleys, and counterweights for raising the lamp for inspection and replacement of carbons.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

(*b*) The lamp shall be surrounded by a framework holding Corex D filters to limit the radiation from the arc essentially to wave lengths found in natural sunlight. Filters and filter frames shall be removable.

(*c*) The lamp and filters shall be centrally located in a drum or circular metal framework covered with sheet metal. The circular framework should carry two sections of rings, each equipped with suitable holders for supporting 32 specimen panels, 3 in. in width and 10½ in. in length. A total of 64 specimen panels shall be accommodated equidistant from the lamp, the distance from the surface of the specimens to the arc to be from 15 to 20 in. The distance of the specimen from the source of light is not critical, but the light should be calibrated from the same distance used for exposure of rubber specimens. Since the degree of deterioration is approximately in proportion to the inverse square of the distance, the rate of deterioration will vary with the distance. In the side of the drum there shall be a sliding door near the bottom to facilitate cleaning or inspecting and changing the test specimens. The top of the drum shall have a cover for shielding the operator from the radiation of the arc. All parts shall be made of metal; the drum, cover, and door shall be made of heavy gage sheet copper tinned on the inside. All structural parts within the drum shall be made of aluminum or rust-proofed steel.

(*d*) The jigs or holders for the test specimens shall be made of aluminum and the rods of monel metal in accordance with the dimensions shown in Fig. 1.

(*e*) The specimen panels shall be rotated at a constant speed about the arc and shall be driven by a constant-speed induction-type motor. The turntable shall rotate one revolution every 2 hr.

(*f*) A water spray shall be provided to thoroughly wet the entire length of each specimen panel for approximately 8 to 10 min. for each revolution. An effective water spray shall be provided for all pressures between 25 and 100 psi. The bottom of the drum shall be equipped with a screened drain to carry away water from the spray.

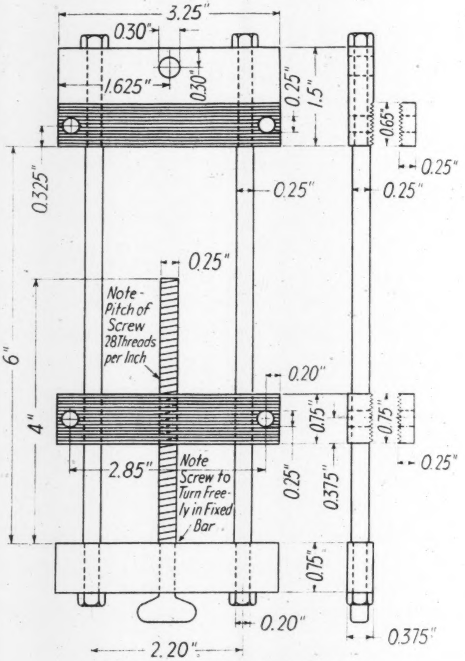

FIG. 1.—Specimen Holder.

All parts, except the rods, made of aluminum; rods made of monel metal.

(*g*) An exhaust fan shall be mounted on the side frame of the testing unit to provide effective ventilation of the arc for the removal of heated air and vapors from the arc. Adequate means shall be provided for lubricating all moving parts.

(*h*) The electrical control equipment shall provide automatic operation of the entire unit and should contain means for keeping the current at the arc constant, insuring constant intensity of radiation. A push-button station shall be provided for starting and stopping the equipment and shall be electrically interlocked with the main line contactor to insure complete electrical isolation of the arc equipment when changing carbons or adjusting the mechanism of the lamp. All control apparatus except the transformer shall be housed in a wall type control cabinet. Toggle switches, pilot light, and ammeter to indicate the arc current shall be mounted on the front of the cabinet.

Calibration of Radiation

4. The light source shall be calibrated at frequent intervals in accordance with the Proposed Method of Calibrating A Light Source Used for Accelerating the Deterioration of Rubber of the American Society for Testing Materials.[2]

Test Specimens

5. Test specimens shall be rectangular strips 2 in. in width and 6 in. in length prepared especially for this test or cut from a product that is to be evaluated. The specimens should have a maximum thickness of 0.100 in. and minimum thickness of 0.045 in. Specimens cut from rubber products may be buffed. Duplicate specimens should be exposed whenever possible.

Procedure

6. (*a*) The test specimens shall be firmly fastened in the jig or holder and elongated to the desired extension, usually 20 per cent, by means of the adjusting screw, Fig. 1. A thermometer shall be mounted adjacent to the specimens in order that the temperature may be observed. The specimen shall then be exposed to the light source until a dosage or total exposure equivalent to the decomposition of the desired number of milligrams of anhydrous oxalic acid as determined by the calibration procedure has been reached. The specimen shall then be removed from the testing unit and examined for degree and number of cracks and for evidence of crazing.

(*b*) A test for the presence in the testing unit of ozone adjacent to the exposed specimens shall be made by exposing a small filter paper previously dipped in starch – potassium iodide solution. Ozone, if present, will cause the test paper to turn blue. The length of time required for the formation of a deep blue coloration may be taken as an indication of the quantity of ozone present. The test paper shall be shielded from the light source.

(*c*) *Tension Tests.*—When the specimen is removed from the jig or holder, two test specimens shall be prepared from it in accordance with the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials.[3] The tensile strength and ultimate elongation shall be determined in accordance with the Tentative Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials.[4] Duplicate unexposed specimens shall be tested for comparison at the same time as the exposed specimens are tested.

Report

7. The report shall include the following:

(*1*) Description and dimensions of the specimens,

(*2*) The number of cracks and degree of crazing or cracking,

(*3*) The intensity of the light source expressed as the number of milligrams of anhydrous oxalic acid decomposed per square decimeter per minute,

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[2] See p. 385.

[3] 1939 Book of A S.T.M. Standards, Part III, p. 331.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 320.

(*4*) The total exposure expressed in equivalents of the number of milligrams of anhydrous oxalic acid decomposed at the rate determined by the average intensity of exposure multiplied by the total length of time of exposure,

(*5*) Tensile strength in pounds per square inch before and after exposure,

(*6*) Elongation at break before and after exposure,

(*7*) The percentage loss in tensile strength as a result of exposure,

(*8*) The percentage loss in elongation at break as a result of exposure,

(*9*) Indication of ozone, and

(*10*) Temperature during exposure to the light source.

APPENDIX II

PROPOSED METHOD OF CALIBRATING A LIGHT SOURCE USED FOR ACCELERATING THE DETERIORATION OF RUBBER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method is intended primarily for use in measuring the intensity of radiation from the light source used in connection with the Proposed Method of Test for Resistance to Accelerated Light Aging of Rubber Compounds.[2] It may also be used for comparing light sources and for measuring and integrating the intensity or total exposure from other light sources which are intermittent or variable in intensity, such as sunlight.

Type of Test

2. This method is based on the fact that oxalic acid in the presence of uranyl sulfate in aqueous solution is photochemically decomposed by light, essentially at a rate in direct proportion to the intensity and total radiation of the light source to which the solution is exposed over the wave length range used for the test. The test method involves the exposure of a standardized oxalic acid - uranyl sulfate solution in a quartz cell to the radiation to be evaluated, followed by a determination of the quantity of oxalic acid decomposed during a measured time period of exposure.

Apparatus

3. (*a*) *Actinometer Cell.*—The actinometer cell (optical) shall be made of thin, clear, fused quartz plates, free from impurities. The wall thickness shall be 1.5 mm., the surfaces ground and highly polished. The internal dimensions of the cell shall be 3 by 4 cm. by 6 cm. in depth with open top. An alternative or working cell in the form of a molded quartz bottle about 75 mm. in diameter by 10 mm. in thickness may also be used, provided it has been calibrated in terms of an optical cell as described above and the correction factor, if any, applied. The cells described are illustrated in Fig. 1.

(*b*) *Cell Enclosure.*—The cell used, whether of the optical or alternative type, shall be enclosed in a lightproof box coated on the inside with a highly light absorbent black paint. The cell shall be totally covered by the enclosure except for one face which shall be exposed through an opening in the enclosure conforming to the full width and depth of the solution in the cell. Cells so mounted are shown in Fig. 2.

Solutions Required

4. (*a*) *Evaluating Solution.*—Prepare an evaluating solution containing 3.16 g. of $KMnO_4$ per liter. After dissolving the $KMnO_4$ in distilled water, store the

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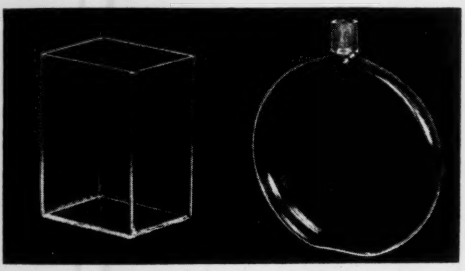
[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

[2] See p. 381.

solution in the dark for at least one week and then filter through a $\frac{1}{8}$-in. thickness of fibrous glass filter cloth, and place the solution in a blackened automatic dispensing burette. Standardize the evaluating solution against sodium oxalate in accordance with the procedure by Kolthoff and Furman.[3]

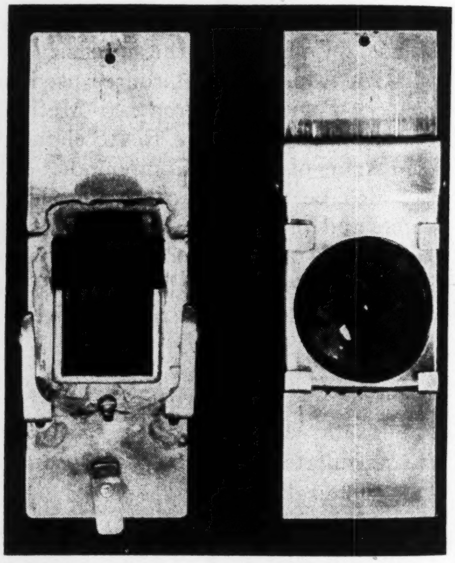
(*b*) *Actinometer Solution.*—Dissolve 6.30 g. of c.p. oxalic acid crystals and 4.20 g. of c.p. uranyl sulfate in distilled water and dilute to 1 liter. This solution will be 0.1 *N* with respect to oxalic acid. Immediately after preparation, transfer the solution to the reservoir of a Machlett type automatic dispensing burette as shown in Fig. 3. The reservoir and glass tubes of the burette should be protected from light by a heavy coating of black enamel. Standardize the actinometer solution as follows: Accurately measure a 50-ml. portion into a 200-ml. tall-form beaker, dilute with 20 to 25 ml. of distilled water, and acidify with 5 ml. of diluted H_2SO_4 (1:3). Cover with a suitable crystallizing dish placed in an inverted position on the beaker, and heat the solution to 95 C. in a lightproof water bath. When the solution reaches 95 C., transfer the beaker to an open glass water bath at the same temperature and resting on a flat, white-glass base. Standardize the actinometer solution against the evaluating solution at 95 C. by addition of the latter from an automatic dispensing burette while stirring constantly. The first few drops of evaluating solution should be added at intervals of several seconds. The end point shall be taken as the change to orange color which persists for 30 sec. while stirring.



Optical Cell. Alternative Cell.

FIG. 1.—Actinometer Cells.

(*c*) Whenever a fresh solution is added to either burette, or if either solution should stand for 60 days or more, restandardize the solutions.



Optical Cell with Filter Glass. Alternative Cell, Filter Glass Not Shown.

FIG. 2.—Actinometer Cells in Housings.

CALIBRATION OF THE LIGHT SOURCE

Procedure

5. (*a*) A suitable quantity of actinometer solution shall be accurately measured into the actinometer cell from the dispensing burette holding the solution. For the rectangular (optical) cell, 50 ml. of solution shall be used. The exposed area in this cell will be approximately 0.16 sq. dm. The alternative

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[3] "Volumetric Analysis," Vol. II, pp. 282–285, John Wiley and Sons, New York, N. Y. (1929).

cell shall always be filled with the same amount of actinometer solution. The quantity shall be such that the solution does not extend excessively into the neck of the bottle or cell but is just sufficient to prevent the formation of an air bubble when the cell is inclined at an angle of 45 deg. Care shall be taken to exclude all stray light from the solution while transporting it to and from the light source under test by enclosing the cell in a suitable lightproof box. The cell shall be mounted on a rubber specimen exposure rack in the testing unit and exposed for a suitable period of time to the light source under normal operating conditions of the testing unit. The time of exposure of the actinometer solution shall not be in excess of that required for the decomposition of 30 per cent of the oxalic acid present.

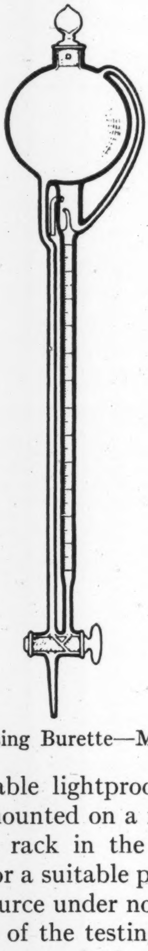

FIG. 3.—Dispensing Burette—Machlett Type.

(*b*) At the end of the period of exposure, the actinometer solution shall be emptied into a 200-ml. tall-form beaker, the cell rinsed with 25 ml. of distilled water, and the solution and wash water acidified with 5 ml. of diluted H_2SO_4 (1:3) per 50 ml. of original undiluted actinometer solution. A blank test shall be made under the same conditions, except that the actinometer solution shall not be exposed to the light source, and the decomposition values shall be adjusted accordingly.

(*c*) The method as described is sufficient for evaluation of the radiation from a standardized light source. Since it is believed that the spectral range of wave lengths between 295 and 335 mμ are particularly instrumental in the deterioration of rubber, it may be desired to measure the intensity in that range alone. If this is desired, the procedure described in Section 7 (*b*) shall be followed.

Calculations

6. The light intensity, in terms of milligrams of oxalic acid decomposed per square decimeter, shall be calculated as follows:

$$\text{Intensity} = \frac{(A - B) \times C}{D \times E}$$

where:

A = milliliters of evaluating solution equivalent to the number of milliliters of unexposed actinometer solution used in the test,

B = milliliters of evaluating solution required for titration of the actinometer solution exposed in the test,

C = milligrams of oxalic acid equivalent to 1 ml. of evaluating solution,

D = time of exposure in minutes, and

E = exposed area in square decimeters of solution in cell.

NOTE: *Example.*—As an illustration, where 50 ml. of evaluating solution (1 ml. = 4.5 mg. of oxalic acid) is the equivalent of the number of milliliters of unexposed actinometer solution used in the test, and 35 ml. of evaluating solution are required for titration of the actinometer solution exposed in the test, the exposed area of the solution in the cell being 0.16 sq. dm., the time of exposure 100 min., the light intensity is calculated as follows:

$$\text{Intensity} = \frac{(50 - 35) \times 4.5}{100 \times 0.16}$$
$$= 4.218 \text{ mg. of oxalic acid per square decimeter per minute.}$$

MEASUREMENT OF TOTAL EXPOSURE AND COMPARISON OF LIGHT SOURCES

Procedure

7. (*a*) This method may be used to determine the exposure to other light sources, including sunlight. Where exposure is intermittent or of variable intensity, the total or integrated exposure or "dosage" only is determined and expressed in terms of milligrams of oxalic acid decomposed per square decimeter.

(*b*) Since it is believed that the wave lengths of light which cause the greatest deterioration of rubber are below 335 mμ, it is desirable in comparing the effectiveness of various light sources to make the comparison over the range of 295 to 335 mμ. This may be accomplished by determining the difference in amount of actinometer solution decomposed in two exposures of equal time and distance with each light source shielded, as follows:

(*1*) Behind 2 mm. of clear Corex D glass which transmits the entire range from 295 mμ to the infra red, and

(*2*) Behind several millimeters of ordinary clear window glass which cuts off radiation near 335 mμ.

Since most accelerated light testing units are provided with suitable filters designed to cut off all radiation below 295 mμ, the actinometer solution in the cell may be exposed directly without interposition of filters. A suitable glass of known characteristics for the second exposure is No. 014 Clear Blue Fluorescing which has approximately 60 per cent transmission at 335 mμ. When using a filter such as the No. 014 glass, a factor of 1/0.92 shall be used in order that the readings obtained from unfiltered and filtered light will be on a comparative basis because of the approximately 8 per cent loss due to reflection. It is desirable to express the difference of intensity in terms of milligrams of oxalic acid per square decimeter per minute.

Calculations

8. The following example will illustrate the method of comparative evaluation of two light sources:

	Oxalic Acid Decomposed Intensity (mg. per sq. dm. per min.)	
	Light Source No. 1	Light Source No. 2
(*a*) Actinometer cell	10.00	5.00
(*b*) Cell behind No. 014 glass color filter, 60 per cent transmission at 335 mμ	8.28	3.68
(*c*) Corrected for surface reflection loss of 8 per cent ($b \times 1/0.92$).	9.00	4.00
(*d*) Evaluation of light source in the range 295 to 335 mμ ($a - c$)	1.00	1.00

While in the above example the total intensity of light source No. 1 is twice that of light source No. 2 as indicated by decomposition of oxalic acid, in the range of intensity which is instrumental in causing the deterioration of rubber the light sources are of equal intensity.

APPENDIX III

PROPOSED METHOD OF TEST FOR TEAR RESISTANCE OF VULCANIZED RUBBER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method describes a procedure which is widely used for determining tear resistance of the usual grades of soft vulcanized rubber such as tire tread, carcass, and inner tube compounds, or those used in most mechanical rubber goods. It does not apply to the testing of material ordinarily classed as hard rubber. Since tear resistance is affected to a large degree by mechanical fibering of the rubber under stress as well as by stress distribution, speed of stretching, and size of specimen, the results obtained in a tear resistance test can only be regarded as a measure of the resistance under the conditions of that particular test and not necessarily as having any relation to service value. The method is useful therefore only for laboratory comparisons and is not applicable for service evaluations, except when supplemented by additional tests, nor for use in purchase specifications. The significance to be attached to the results can only be determined by each laboratory for its particular problem.

Apparatus

2. (*a*) Tear resistance shall be measured on a power-driven apparatus, preferably of the inclination-balance or pendulum type. A spring-balance type of apparatus may be used if provided with a device that will indicate the actual maximum load at which rupture took place and if provision is made to prevent recoil of the spring. The machine shall conform to the following requirements:

(*b*) The applied load as indicated by a dial or scale shall be accurate within plus or minus 1 per cent.

(*c*) The indicator shall remain at the point of maximum load after rupture of the test specimen.

(*d*) The grips which hold the specimen in the testing machine shall have jaws of the narrow cam or pincer type.

(*e*) The rate of travel of the power actuated grip shall be 20 in. per min. and shall be uniform at all times.

(*f*) The testing machine shall be calibrated in accordance with the provisions of Section 3.

(*g*) The testing machine may be equipped with a dynamometer head of the compensating type for convenience in eliminating calculations.

Calibration of Apparatus

3. The testing machine shall be calibrated by dead-weights applied in an ascending order while the machine is otherwise arranged in an entirely similar manner to that used when testing material. In each case, when additional calibrating weight is added the weight lever of the machine shall be moved to

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber Products.

zero position and allowed to swing slowly upwards to the point where it will go no further. The dial of the machine should then clearly indicate the amount of the weight applied.

Preparation of Sample

4. Except as may be otherwise specified in this method, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials[2] shall be complied with and are made a part of this method.

Die for Test Specimens

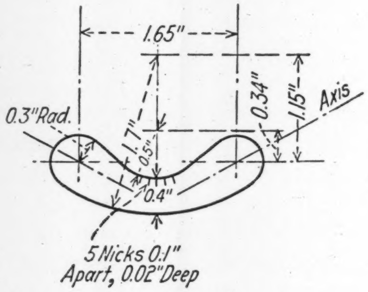
5. Unless otherwise prescribed in the specifications for the material being tested, the specimens for tear resistance tests shall be stamped out with a steel die. The die shall be kept sharp and free from nicks to avoid leaving ragged edges on the rubber. Cutting may be facilitated by wetting the rubber surface and the cutting edges of the die. The rubber shall be rested on a smooth, slightly yielding surface that will not injure the blade. Lightweight cardboard or a piece of leather belting is suitable. Care shall be taken that the cut edges are perpendicular to the other surfaces of the specimen and have a minimum of concavity.

NOTE.—It is a well-known fact that vulcanized rubber exhibits a grain effect which influences its physical properties. In the case of heavily loaded compounds, the effect may produce a pronounced difference in physical properties. Usual practice is to test with the grain running the long way of the specimen, and it is assumed that, unless otherwise specified, all test specimens are to be prepared in this manner. Where grain effects are significant and are to be evaluated, two sets of test specimens should be cut from the sample at right angles to each other.

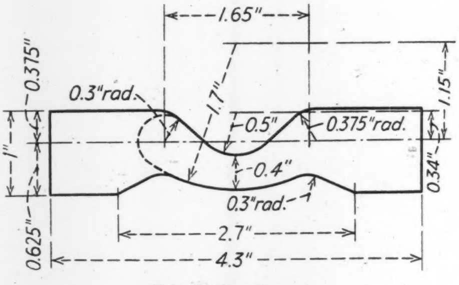
Test Specimens

6. (*a*) The test specimens shall conform in shape to die A or B as shown in Fig. 1, and shall not vary by more than plus or minus 0.01 in. from the dimensions shown. The thickness shall not fall outside the limits 0.07 to 0.11 in.

(*b*) The thickness of the specimen shall be measured with a micrometer graduated to 0.001 in., having a presser foot 0.25 ± 0.01 in. in diameter exerting a total force of 3.0 ± 0.1 oz. The load shall be applied by means of a weight.



Die A Specimen.



Die B Specimen.

FIG. 1.—Specimens for Tear Test.

(*c*) The five slits in the specimen shall be cut with extreme care and accuracy. A device (Fig. 2) which holds the specimen stretched so that the inner curved edges are drawn out straight, and which is provided with suitable guides to control the depth and centering of the slits shall be used. The cutting shall be done by safety razor blades clamped in a holder exactly 0.1 in. apart. The extension of the blades beyond the surface of the knife holder shall be regulated by a depth gage block to produce slits 0.02 in. in depth. In cutting the slits, the specimens held in the stretching device shall first be lubricated with soap solution, and the knives shall then be drawn back and forth through the guides. Equipment suitable for use with die A specimen is shown in Fig. 2.

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[2] 1939 Book of A.S.T.M. Standards, Part III, p. 331.

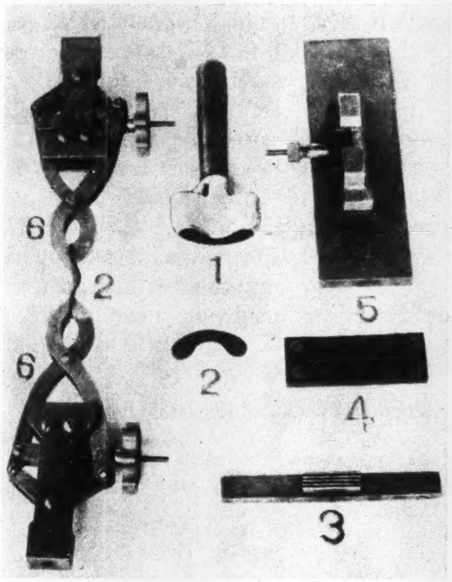

FIG. 2.—Apparatus for Preparing Die A Specimen.

1. Hand die for cutting specimen.
2. Rubber specimen cut with die A.
3. Knife holder for cutting slits.
4. Depth gage for fitting blades in holder.
5. Vise for holding specimen while cutting slits.
6. Special testing machine clamps for specimens cut with die A.

Number of Test Specimens

7. (*a*) Three specimens per sample shall be tested for tear resistance. In case the tear resistance fails to check within 20 per cent of the highest value obtained, additional specimens shall be tested until two or more such check results are obtained. The final value shall be the average of the check tests, discarding the determinations on specimens that are outside the above limit.

(*b*) When minimum requirements are established, as in testing for compliance with a particular specification, only one specimen shall be tested for tear resistance. If the result is below the specification requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirements, the sample shall be considered to have failed to conform to the specifications.

Procedure

8. The specimen for test shall be clamped in the jaws of the testing machine, care being taken that the jaws grip the specimen so that the axes shown in Fig. 1 are in line with the direction of the application of the load. Increased stretch is thus given to the inner curved edge. The "bite" of the jaws shall be at the center of the enlarged ends of the specimen. The load shall then be applied with a lower jaw speed of 20 in. per min. After rupture of the specimen, the breaking load in pounds shall be noted from the dial or scale and recorded together with the average thickness of the specimen.

Calculation

9. The resistance to tear shall be calculated from the maximum load registered by the testing machine and the average thickness of the specimen, and shall be expressed as the pull in pounds required to tear a specimen 1 in. in thickness.

NOTE: *Example.*—If a load of 36 lb. is necessary to tear a specimen 0.082 in. in thickness, the tear resistance would be $= \frac{36.0}{0.082} =$ 439 lb. per in.

Report

10. The report shall include the following:

(*1*) Results calculated in accordance with Section 9,

(*2*) All observed and recorded data on which the calculations are based,

(*3*) Date of test and date of vulcanization of rubber, if known,

(*4*) Test room temperature,

(*5*) Type of testing machine used, and

(*6*) Designation of die used in preparing the test specimens.

REPORT OF COMMITTEE D-12
ON
SOAPS AND OTHER DETERGENTS

Committee D-12 on Soaps and Other Detergents held two well-attended and interesting meetings during the year: one on November 2 and 3, 1939, and the second on March 11 and 12, 1940, both at the Hotel New Yorker, New York City.

At the spring meeting W. D. Pohle, of the Naval Stores Research Division of the U. S. Bureau of Agricultural Chemistry and Engineering, gave a very interesting talk descriptive of his work on rosin and rosin soaps.

The following new members have been elected to Committee D-12 during the year:

L. F. Hoyt, National Aniline and Chemical Co.,
H. G. Arlt, Bell Telephone Laboratories, Inc.,
M. H. Gurley, Jr., Gustavus J. Esselen, Inc., and
Katharine Fisher, Good Housekeeping Inst

In addition, H. L. Jones of E. I. du Pont de Nemours and Co. replaced Samuel Lenher; Charles Schwartz of Calgon, Inc. replaced B. F. Gilmore; and D. D. Rubek of Anderson-Prichard Oil Corp. replaced J. K. Stewart. E. T. Marceau has resigned due to a change of position; W. M. Scott has accepted a position with the Southern Regional Research Laboratories, New Orleans, La., but will retain his membership on Committee D-12.

The committee voted to omit the fall meeting in 1940, with the understanding that the chairmen of the subcommittees would endeavor to have the sections complete their work more fully by correspondence. If that is done, the committee, at the 1941 spring meeting, would be able to ratify the completed work and prepare the report for the annual meeting.

On the recommendation of Subcommittee IV, Committee D-12 approved the preparation of a paper by the chairman, with the assistance of a number of the members, on the manufacture and testing of soaps and other detergents. This paper is published in the ASTM BULLETIN, No. 107, December, 1940, p. 9.

The election of officers and members of the advisory committee for the ensuing term of two years has resulted in the re-election of the present incumbents.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following four new specifications be accepted for publication as tentative. These specifications were prepared in response to requests received by the committee in view of the large consumer use of these products:

Tentative Specifications for:[1]

Olive Oil Solid Soap,
Salt-Water Soap,
Tetrasodium Pyrophosphate (Anhydrous), and
Sodium Sesquisilicate.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following tentative standards be revised and continued as tentative:

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[1] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 508, 512, 515, and 514, respectively.

Tentative Specifications for Palm Oil Bar Soap (D 535 – 39 T),[2] covering the addition of requirements for palm oil solid soap (blended type);[3]

Tentative Methods of Sampling and Chemical Analysis of Special Detergents (D 501 – 39 T),[4] covering the addition of a new method of sampling and analysis of sodium sesquisilicate;[5] and

Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 39 T),[6] covering a revision of five definitions (alkaline detergent, break soap powder, builder, built soap, washing), and the omission of the definition for scouring; also the addition of new definitions for blended (palm oil) soap, filler, pure (palm oil) soap, and straight soap.[7]

III. TENTATIVE REVISION OF STANDARD

The committee recommends that the revision, as given in the Appendix to this report, of the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 39)[8] be accepted for publication as tentative.[9]

IV. REVERSION OF STANDARD TO TENTATIVE

The committee recommends that the Standard Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 39)[10] be reverted to tentative with a revision in the definitions for soap and anhydrous soap, the definitions to be included with the new and revised tentative definitions mentioned earlier in this report. The complete list of definitions is appended hereto.[7]

V. EDITORIAL CHANGES IN STANDARDS

The committee recommends as an editorial change that "anhydrous soap" be changed to read "anhydrous soap content" in the table of chemical requirements and in Section 4 of the following specifications:

Standard Specifications for:[11]

Milled Toilet Soap (D 455 – 39),
Chip Soap (D 496 – 39)
Ordinary Bar Soap (D 497 – 39),
Powdered Soap (Nonalkaline Soap Powder) (D 498 – 39),
White Floating Toilet Soap (D 499 – 39),

Tentative Specifications for:[12]

Built Soap (Powdered) (D 533 – 39 T),
Soap Powder (Alkaline Soap Powder) (D 534 – 39 T),
Palm Oil Bar Soap (D 535 – 39 T), and
Palm Oil Chip Soap (D 536 – 39 T).

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Olive Oil Solid Soap[a]	33	6	2
Spec. for Salt-Water Soap	36	0	5
Spec. for Tetrasodium Pyrophosphate (Anhydrous)	38	0	3
Spec. for Sodium Sesquisilicate	38	0	3
II. REVISION OF TENTATIVE STANDARDS			
Spec. for Palm Oil Bar Soap (D 535 - 39 T)[b]	33	5	3
Sampling and Chemical Analysis of Special Detergents (D 501 - 39 T)	38	0	3
Definitions of Terms Relating to Soaps and Other Detergents (D 459 - 39 T)	38	3	0
III. TENTATIVE REVISION OF STANDARD			
Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 39)	35	2	4
IV. REVERSION OF STANDARD TO TENTATIVE			
Definitions of Terms Relating to Soaps and Other Detergents (D 459 - 39), as revised	40	0	1

[a] The classified vote on the proposed Tentative Specifications for Olive Oil Solid Soap was as follows: Affirmative: 12 producers, 13 consumers, 8 general interests; negative: 5 producers, 0 consumers, and 1 general interest.

[b] The classified vote on the proposed revision of the Tentative Specifications for Palm Oil Bar Soap (D 535 - 39 T) was as follows: Affirmative: 13 producers, 12 consumers, 8 general interests; negative: 4 producers, 0 consumers, 1 general interest.

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[2] 1939 Book of A.S.T.M. Standards, Part III, p. 1061.

[3] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 510.

[4] 1939 Book of A.S.T.M. Standards, Part III, p. 1070.

[5] See p. 398.

[6] 1939 Book of A.S.T.M. Standards, Part III, p. 1089.

[7] These revised definitions were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 521.

[8] 1939 Book of A.S.T.M. Standards, Part III, p. 487.

[9] See p. 398.

[10] 1939 Book of A.S.T.M. Standards, Part III, p. 518.

[11] 1939 Book of A.S.T.M. Standards, Part III, pp. 477 to 486, incl.

[12] *Ibid.*, pp. 1061 to 1067, incl.

In the Standard Method of Test for Particle Size of Soaps and Other Detergents (D 502 – 39)[13] following the first sentence in Sections 4 and 5, add a parenthetical sentence to read as follows: "Other size sieves may be used when required by the individual specifications."

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 62 members; 41 members returned their ballots, with the results shown in Table I.

Activities of Subcommittees

Subcommittee I on Methods of Testing (J. B. Crowe, chairman):

Section A on Soaps (M. L. Sheely, chairman) has completed a revision of the titer test in the methods of chemical analysis of soaps and soap products (D 460 – 39) to include a new thermometer and a complete rewriting of Sections 35 and 36 on apparatus and procedure; also minor revisions in Sections 16, 17, and 34. The committee has approved the use of sieves of other than the three sizes now specified in the Methods of Test for Particle Size of Soaps (D 502 – 39).

Since the method appears promising, the section is now working on the determination of carbon dioxide in soaps by the Hitchcock-Divine method, having appointed a special committee to study the procedure in detail. Work is also under way on the determination of pyrophosphate in soaps.

This section is practically identical in personnel with the soap committee of the American Oil Chemists' Society and it also works in close cooperation with the soap committee of the American Chemical Society; thus the test methods of the three societies should be identical.

Section B on Sulfonated Detergents (Ralph Hart, chairman) has not been so active this past year but it expects soon to undertake some cooperative work on new methods of test developed by the chairman.

Section C on Dry Cleaning Detergents (G. P. Fulton, chairman) has a very difficult assignment but is making definite progress.

Section D on Special Detergents (W. H. Koch, chairman) has considered methods of analysis of tetrasodium pyrophosphate and sodium orthosilicate. The previously submitted method for tetrasodium pyrophosphate was revised, and three samples of this material were distributed to the committee members for cooperative work, the results of which indicated that with a further slight modification the method can be made entirely satisfactory. This revision and three additional samples will be supplied to the members in the near future for additional tests to confirm this opinion. This work will also include confirmation of the methods of manufacture of c.p. tetrasodium pyrophosphate for standardization purposes.

The method of analysis of sodium orthosilicate under consideration in the section will require considerable revision, after which it will again be submitted for comment and criticism at an early date. Considerable discussion has centered about the desirability of presenting this method to the Society for publication as tentative. Proof of any nature seems to be lacking that there is actually such a compound as sodium orthosilicate; it has almost definitely been shown to be a mixture of sodium metasilicate and caustic soda. The section will, however, proceed to develop a method of analysis for this product and Committee D-12 can later determine whether or not it wishes to approve the name "sodium orthosilicate" and to include it in the specifications and methods of analysis of special detergents.

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[13] 1939 Book of A.S.T.M. Standards, Part III, p. 509.

Subcommittee II on Specifications (F. W. Smither, chairman):

This subcommittee held two meetings during the year. The various sections of Subcommittee II have been quite active, but the time and study required for the difficult assignments now on hand have necessarily limited the number of definite recommendations at this time.

Section A on Textile Soaps (C. L. Nutting, chairman) and Section C on Straight Soaps (Israel Katz, chairman) have worked together during the past year in order to facilitate accomplishment by eliminating duplication of effort and by mutual assistance. The Tentative Specifications for Palm Oil Bar Soap (D 535 – 39 T) have been amended to apply to two types, pure and blended, and the title changed to Specifications for Palm Oil Solid Soap. The revised specifications are accordingly being recommended for publication as tentative, as mentioned earlier in this report.

The section is recommending that the Tentative Specifications for Palm Oil Chip Soap (D 536 – 39 T), with certain editorial corrections, be continued as tentative. Also, Specifications for Olive Oil Solid Soap are being submitted for publication as tentative.

Section B on Built Soaps (Frederick Krassner, chairman) is recommending that the Tentative Specifications for Soap Powder (Alkaline Soap Powder) (D 534 – 39 T) and for Built Soap (Powdered) (D 533 – 39 T) be continued as tentative with a minor editorial change.

Proposed Specifications for Salt-Water Soap are presented for publication as tentative as mentioned earlier in the report. The section is submitting as information, in order to elicit comment and criticism, Proposed Specifications for Detergent Soap Powder and for Grit Cake Soap, which appear as Appendices II and III, respectively.

Section D on Dry Cleaning Detergents (G. P. Fulton, chairman) has reviewed the literature on this subject and has concluded that specifications for dry cleaning soaps could best be formulated at this time on the basis of physical characteristics, and perhaps on the performance of the soap when used in conjunction with a pressure filter. On this basis, dry cleaning soaps could be divided into several classes, such as standard paste-type soap, filterable paste-type soap, liquid soaps, and filterable liquid soaps. The section will continue its work on this difficult problem.

Section E on Sulfonated Detergents (J. B. Crowe, chairman) has continued the cooperative laboratory studies on washing tests, and is preparing an analysis and summary of the results obtained. The formulation of definite specifications for products in this field is a difficult and tedious undertaking. The work will be continued.

Section F on Special Detergents (C. C. Zeigler, chairman) has prepared Specifications for Tetrasodium Pyrophosphate (Anhydrous) and for Sodium Sesquisilicate, which are being recommended for publication as tentative, as mentioned earlier in this report. This section is also considering the feasibility of preparing specifications for sodium orthosilicate.

Section G on Metal Cleaners (J. C. Harris, chairman) has made definite progress on a very difficult assignment. The section has formulated methods of test that would be applicable to some types of cleaners, but as such a wide variety of materials are used in metal cleaning compositions, it was concluded that specifications based solely on chemical analysis would indicate only a few of the desirable properties that a given preparation should possess. The section is therefore working on certain performance tests that may ultimately form the basis for specifications.

Subcommittee III on Nomenclature and Definitions (C. A. Marlies, chairman):

This subcommittee is recommending that the Standard Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 39) be reverted to tentative and that the definitions for soap and anhydrous soap (to be changed to anhydrous soap content) be revised. Also, in the Tentative Definitions D 459 – 39 T the definitions for alkaline detergent (to be changed to alkali detergent) break soap powder (to be changed to detergent soap powder), builder, built soap, and washing are being revised; the definition for scouring is being omitted; and the term soapy alkaline detergent is being changed to soapy alkali detergent. In addition, definitions of the following four terms are being presented: blended (palm oil) soap, filler, pure (palm oil) soap, and straight soap. The complete list of definitions is appended hereto.[11]

This subcommittee has also recommended that the term "anhydrous soap" be changed to read "anhydrous soap content" in the five standard and four tentative specifications for soaps, listed earlier in this report.

Subcommittee IV on Papers and Publications (W. H. Koch, chairman):

This subcommittee reports progress on the preparation of a monograph on the testing and use of soaps and other detergents. The section covering caustic soda in the proposed monograph on alkali detergents was thoroughly criticized with the result that it will be edited, rewritten, and again submitted for approval. At the same time various members of the committee will be asked to draft a similar article on some one of the alkali detergents which Committee D-12 has so far considered. It is expected that by the next meeting of the committee these drafts will be in such shape that they can be rewritten and put in final form.

The paper submitted by Mr. Trevithick on the activities of Committee D-12 has been approved by Subcommittee IV. It is felt that the publication of this paper will go far in acquainting the Society with the work of the committee.

This subcommittee has approved for publication as information the two proposed specifications submitted by Section B of Subcommittee II covering Detergent Soap Powder and Grit Cake Soap which appear in Appendices II and III.

This report has been submitted to letter ballot of the committee which consists of 62 members; 41 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. P. TREVITHICK,
Chairman.

B. S. VAN ZILE,
Secretary.

APPENDIX I

PROPOSED REVISIONS IN STANDARDS FOR SOAPS AND OTHER DETERGENTS

In this Appendix are given proposed revisions in one standard method and one tentative method for the analysis of soaps and other detergents which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the standard appears in its present form.

Revision of Tentative Standard

Tentative Methods of Sampling and Chemical Analysis of Special Detergents (D 501 - 39 T):[1]

Section 6.—In this section and in the heading preceding it, add the words "and sodium sesquisilicate" after "sodium metasilicate."

New Section.—Add the following as a new Section 10, renumbering the present Section 10 as 11:

10. *Sodium Sesquisilicate,* $3Na_2O \cdot 2SiO_2 \cdot 11H_2O$.—If the ratio of the percentage of SiO_2 divided by the percentage total alkalinity as Na_2O is less than 0.646, calculate the percentage of sodium sesquisilicate as follows:

$$3Na_2O \cdot 2SiO_2 \cdot 11H_2O\text{, per cent} = \text{total } SiO_2\text{, per cent} \times 4.20$$

If the ratio is greater than 0.646, calculate the percentage of sodium sesquisilicate as follows:

$$3Na_2O \cdot 2SiO_2 \cdot 11H_2O\text{, per cent} = \text{total } Na_2O\text{, per cent} \times 2.71$$

New Section.—As a new Section 12, add the following procedure for loss on ignition of sodium sesquisilicate:

12. *Loss on Ignition of Sodium Sesquisilicate.*—Weigh about 2 g. of sand in a clean platinum crucible with a tight-fitting lid, and ignite to constant weight. Weigh about 2 g. of the sodium sesquisilicate in the crucible, and heat with a low flame until the silicate is melted Increase the heat gradually as the water is driven off, care being taken to prevent spattering. Ignite to constant weight. Cool in a desiccator, and weigh. Calculate the loss on ignition as follows:

$$\text{Loss on ignition, per cent} = \frac{\text{loss in wt.}}{\text{wt. of sample}} \times 100$$

Tentative Revision of Standard

Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 39):[2]

Section 16.—Add a note at the end of this section to read as follows:

NOTE.—In the analysis of soaps known to contain little or no alkaline salts, it is unnecessary to filter the hot alcoholic soap solution as described in Section 15. However, the filtration should be carried out in all cases where alkaline salts such as silicates, phosphates, borates, and similar salts are present, since these are known to affect the free alkali determination. The presence of carbonates up to 0.5 per cent as Na_2CO_3 does not appreciably affect this determination and filtration may be omitted if the carbonate determination on a separate sample is not in excess of this amount.

Tentative revision, submitted June, 1940.

Section 17.—Change the first sentence to read as follows by the addition of the italicized words:

Proceed as in the determination of matter insoluble in alcohol (starting with a fresh sample of soap *and omitting the drying and weighing of matter insoluble in alcohol*) (Section 15).

Tentative revision, submitted June, 1940.

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[1] 1939 Book of A.S.T.M. Standards, Part III, p. 1070.

[2] 1939 Book of A.S.T.M. Standards, Part III, p. 487.

Section 34.—In the first paragraph change the next to the last sentence to read as follows by the addition of the italicized words:

> After the last washing, remove the last traces of water from the beaker with a pipette, filter the fatty acids through one or two thicknesses of filter paper, and dry at a temperature of 105 C. for 45 to 60 min. *or heat rapidly to 130 C. and allow to cool. Do not hold at 130 C., but if water is present, decant the clear fatty acids into another beaker, and again reheat them momentarily to 130 C.*
>
> *Tentative revision, submitted June, 1940.*

Sections 35 and 36.—Change the description of the apparatus and procedure for the titer test from its present form to read as follows:

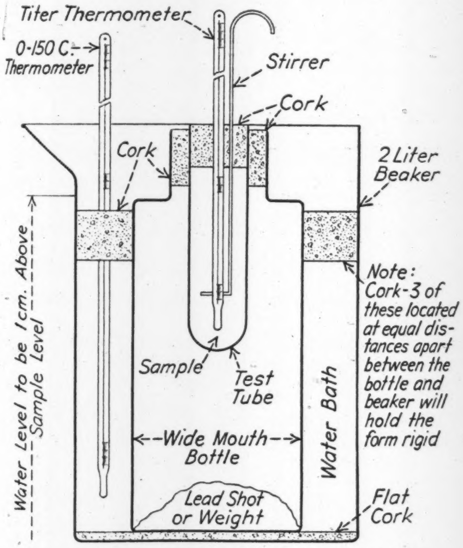
Titer Thermometer
0-150 C. Thermometer
Stirrer
Cork
Cork
2 Liter Beaker
Water Level to be 1 cm. Above Sample Level
Note: Cork-3 of these located at equal distances apart between the bottle and beaker will hold the form rigid
Sample
Test Tube
Water Bath
Wide Mouth Bottle
Lead Shot or Weight
Flat Cork

FIG. 1.—Apparatus Assembly for Titer Test.

35. *Apparatus.*—The apparatus, Fig. 1, shall consist of the following:

(*a*) *Water Bath.*—A 2-liter Griffin low-form beaker.

(*b*) *Wide-Mouth Bottle.*—A wide-mouth 450-ml. bottle, 190 mm. in height, having a neck 38 mm. in inside diameter, fitted with a cork to carry the test tube (Paragraph (*c*)) and with sufficient weights or lead in the bottom of the bottle to hold it steady when placed in the water bath.

(*c*) *Test Tube.*—Test tube 100 mm. in length, and 25 mm. in diameter with or without rim. The tube may have an etched line extending around it at a distance of 57 mm. from the bottom to indicate the height to which it is to be filled.

(*d*) *Stirrer.*—A stirrer 2 to 3 mm. in outside diameter with one end bent in the form of a loop 19 mm. in diameter. Glass, nichrome, stainless steel, or monel metal wire may be used. The upper end of the stirrer may be formed to accommodate hand stirring or for attachment to a mechanical stirrer.

(*e*) *Laboratory Thermometer.*—A laboratory thermometer having a range of 0 to 150 C.

(*f*) *Titer Test Thermometer.*—An A.S.T.M. Titer Test Thermometer, graduated in Centigrade degrees, having a range of −2 to +68 C. and conforming to the following requirements:

TYPE: Etched stem, glass.

LIQUID: Mercury.

RANGE AND SUBDIVISION: −2 to +68 C. in 0.2 C.

TOTAL LENGTH: 375 to 385 mm.

STEM: Suitable thermometer tubing, plain front or magnifying lens type (red reading mercury preferable but not obligatory), enamel back.
Diameter, 6.0 to 7.0 mm.

BULB: Corning normal or equally suitable thermometric glass.
Length, 15 to 25 mm.
Diameter, not less than 5.5 mm. but not greater than stem.

DISTANCE to −2 C. LINE FROM BOTTOM OF BULB: 50 to 60 mm.

LENGTH OF GRADUATED SCALE: 300 mm. min.

LENGTH OF UNCHANGED CAPILLARY BETWEEN LOWEST GRADUATION LINE AND BULB: 13 mm. min.

EXPANSION CHAMBER: To permit heating to at least 85 C.

LENGTH OF UNCHANGED CAPILLARY BETWEEN UPPERMOST GRADUATION LINE AND EXPANSION CHAMBER: 10 mm. min.

TOP FINISH: Glass ring.

FILLING ABOVE LIQUID: Evacuated, or filled with nitrogen or other suitable inert gas.

GRADUATION: All lines, figures, and letters clear cut and distinct. The whole degree Centigrade lines to be longer than the remaining lines. Graduations to be numbered at each multiple of 2 C.

IMMERSION: 45 mm.

MARKING: "A.S.T.M. Titer Test," a serial number, and the manufacturer's name or trade mark shall be etched on the stem. The words "45-mm.-immersion" and a line 45 mm. above the bottom of the bulb shall be etched on the thermometer.

SCALE ERROR: The error at any point of the scale, when the thermometer is standardized at 45-mm. immersion, shall not exceed 0.2 C.

STANDARDIZATION: The thermometer shall be standardized at intervals of approximately 10 C. for 45-mm. immersion and for an average temperature of the emergent mercury column of 25 C.

CASE: The thermometer shall be supplied in a suitable case on which shall appear the marking: "A.S.T.M. Titer Test, −2 to +68 C. in 0.2 C."

NOTE.—For the purpose of interpreting these specifications, the following description of terms apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage or micrometer.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

36. *Procedure:* (*a*) Transfer the fatty acids (Section 34) when cooled somewhat, to the test tube filling it to the 57-mm. line, and place the tube in the wide-mouth bottle. Set the bottle assembly in the water bath (the water should reach the neck of the bottle), as shown in the accompanying Fig. 1 and adjust the temperature of the bath to 20 C. for fatty acids with titers of 35 C. and over, and to 15 to 20 C. below the expected titer for all fatty acids with titers below 35 C.

(*b*) Insert the titer test thermometer in the center of the sample and suspend it at such a height that the immersion mark coincides with the top of the sample of fatty acids.

(*c*) When the titer test thermometer reads 10 C. above the expected titer, stir with the stirring rod in a vertical manner at the rate of 100 completed up-and-down motions per minute. The stirrer should travel about 3.8 cm. Read the thermometer every 15 sec., and continue stirring until the temperature remains constant (or begins to rise) for 30 sec. Discontinue stirring immediately and note the rise in temperature. Report as the titer of the fatty acids, the highest temperature reached by the thermometer.

(*d*) Duplicate determinations should not differ by more than 0.2 C.

Tentative revision, submitted June, 1940.

APPENDIX II

PROPOSED SPECIFICATIONS FOR DETERGENT SOAP POWDER[1]

These are proposed specifications and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These specifications cover a detergent soap mixture in powder form suitable for uses in which large amounts of alkaline materials are desirable.

General Requirements

2. Detergent soap powder shall be a high-grade, uniformly mixed product, composed of soap and alkaline salts in powdered form. The soap powder shall be of uniform color, free from disagreeable odor, dirt, and other foreign matter. It shall be soluble in water at low wash-wheel temperatures without the formation of difficultly soluble aggregates.

Chemical Composition

3. Detergent soap powder shall conform to the requirements as to chemical composition prescribed in Table I. The percentage of matter volatile at 105 C. shall be calculated on the basis of the soap as received, but all other constituents shall be calculated on the basis of material containing 16 per cent of matter volatile at 105 C.

Basis of Purchase

4. Detergent soap powder is subject to a possible gain or loss of weight, depending on atmospheric or storage conditions, or both, or on packaging as a result of fluctuation in the moisture content. Changes in the moisture content result in a corresponding change in the percentage of total solids or anhydrous soap content, or both.

(*a*) The material shall be purchased by net weight, provided the matter volatile at 105 C. is neither above nor below 13 per cent.

TABLE I.—CHEMICAL REQUIREMENTS.

Moisture and matter volatile at 105 C., max., per cent	16.0[a]
Sum of free alkali and total matter insoluble in alcohol (alkaline salts), max., per cent	60.0
Matter insoluble in water, max., per cent	1.0
Titer of mixed fatty acids prepared from the soap, max	35 C.
Material passing an 105-micron (No. 140) sieve, max., per cent	20.0
Material retained on an 1680-micron (No. 12) sieve, max., per cent	1.5
Rosin acids	none
Anhydrous soap content, per cent	25 to 50

[a] Deliveries which yield more than 16 per cent volatile matter shall be rejected without further test.

(*b*) Deliveries containing more than 16 per cent of matter volatile at 105 C. shall be rejected without further test.

(*c*) On deliveries containing less than 16 per cent of matter volatile at 105 C., settlement shall be made on the basis of a product containing 13 per cent of moisture, that is, 0.87 lb. of nonvolatile matter shall be considered 1 lb. of soap.

(*d*) When the material conforms to these specifications on a calculated 16 per cent moisture and volatile matter basis, the net weight of the material to be paid for shall be calculated as follows:

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[1] These proposed specifications are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Other Detergents.

$$W = \frac{R \times (100 - L)}{87}$$

where:

W = net weight of the material to be paid for on 13 per cent moisture and volatile matter basis,
R = net weight of material, as received, and
L = percentage loss at 105 C.

(*e*) In the event that the calculation under Paragraph (*d*) results in a figure greater than 100 per cent, due to low moisture content at the time of filling, weighing, and packing, the seller waives the right to invoice the purchaser for any amount in excess of 100 per cent.

Methods of Sampling and Analysis

5. (*a*) The material shall be sampled and analyzed in accordance with the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (A. S. T. M. Designation: D 460) of the American Society for Testing Materials.[2] The purchaser reserves the right to use any additional available information to ascertain whether the material conforms to these specifications.

(*b*) The sieve analysis shall be made in accordance with the Standard Method of Test for Particle Size of Soaps and Other Detergents (A. S. T. M. Designation: D 502) of the American Society for Testing Materials.[3]

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[2] 1939 Book of A.S.T.M. Standards, Part III, p. 487.
[3] *Ibid.*, p. 509.

APPENDIX III

PROPOSED SPECIFICATIONS FOR GRIT CAKE SOAP[1]

These are proposed specifications and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These specifications cover grit soap for scouring and scrubbing purposes, of two types as follows:

Type A, for fine work such as glass and enamel, and

Type B, for general scouring and scrubbing.

General Requirements

2. Grit soap shall be well compressed cakes of a satisfactory degree of friability, which shall not be materially affected or lessened after immersion or contact with water. The soap shall be unscented and shall be of a light gray or white color.

Chemical Composition

3. Grit cake soap, as received, shall conform to the requirements as to chemical composition prescribed in Table I.

Basis of Purchase

4. Grit cake soap of both types shall be purchased by net weight.

Methods of Sampling and Analysis

5. (*a*) The material shall be sampled and analyzed in accordance with the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (A. S. T. M. Designation: D 460),[2] of Sampling and Chemical Analysis of Special Detergents (A. S. T. M. Designation: D 501 - 39),[3] and the Tentative Methods of Sampling and Chemical Analysis of Special Detergents (A. S. T. M. Designation: D 501 - 40 T)[4] of the American Society for Testing Materials. The purchaser reserves the right to use any additional available information to ascertain whether the material conforms to these specifications.

TABLE I.—CHEMICAL REQUIREMENTS.

	Type A	Type B
Moisture and matter volatile at 105 C., max., per cent	4.0	5.0
Alkali as alkaline salts (total alkalinity of matter insoluble in alcohol, calculated as Na_2CO_3), max., per cent	1.0	3.0
Free alkali, calculated as NaOH, max., per cent	0.1	0.1
Siliceous matter insoluble in water, per cent	88 to 93[a]	75 to 85[a]
Material passing an 149-micron (No. 100) sieve, per cent	100	100
Material retained on a 74-micron (No. 200) sieve, max., per cent	5.0	...
Rosin, sugar, and foreign matter	none	none
Anhydrous soap content	shall be within 1 per cent of the difference between 100 and the sum of matter volatile at 105 C., insoluble siliceous matter, and alkali as alkaline salts	

[a] Insoluble siliceous material in type A soap shall consist of not less than 90 per cent of ground feldspar; in type B it shall consist mainly of quartz.

(*b*) The sieve analysis shall be made in accordance with the Standard Method of Test for Particle Size of Soaps and Other Detergents (A. S. T. M. Designation: D 502) of the American Society for Testing Materials.[5]

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[1] These proposed specifications are under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Other Detergents.

[2] 1939 Book of A.S.T.M. Standards, Part III, p. 487.

[3] *Ibid.*, p. 515.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 516.

[5] 1939 Book of A.S.T.M. Standards, Part III, p. 509.

REPORT OF COMMITTEE D-13

ON

TEXTILE MATERIALS

Committee D-13 on Textile Materials held two meetings during the year: one on October 18 to 20, 1939, in New York, N. Y., and the other on March 13 and 14, 1940, in Charlotte, N. C. The number of members and guests registered was 138 and 98, respectively. A third meeting will be held in June at Atlantic City, N. J., in conjunction with the annual meeting of the Society.

The executive direction of the committee's affairs required three meetings of the officers and two meetings of the advisory committee. Of the 28 subcommittees or sections which compose the organization of the committee, 24 held sessions at the fall meeting and 17 at the spring meeting. From these facts, together with the many recommendations regarding standards included herein, it is evident that interest in standardization in the textile field is as active and productive as usual.

In March the committee held its first meeting in the South since 1930. Its purpose was to bring the work of Committee D-13 to the attention of a larger number of the technical men of this important textile producing region, and to secure their cooperation in the development and increased use of the textile standards of the American Society for Testing Materials. The committee is indebted to F. S. Mapes, D. E. Truax, and W. B. Hodge who served as a Committee on Arrangements, and to R. W. Philip and B. E. Royal who ably handled the publicity features. The papers presented at this meeting are given in the report of Subcommittee B-6 on Papers.

Only one change has occurred in the membership of the advisory committee. L. H. Nicholson has been appointed chairman of Section II on Yarn, Roving, and Twine of Subcommittee A-8 on Glass Fiber and its Products *vice* S. J. Rosch, resigned.

At the March meeting upon the recommendation of Subcommittee B-4 on Bleaching, Dyeing, and Finishing, W. M. Scott as its chairman and as the representative of the Society on the Inter-Society Color Council, presented to the general session of Committee D-13 the following motion which was approved unanimously and in accordance with the vote is presented here:

> In order to promote uniformity of nomenclature in the presentation of textile color data, Committee D-13 shall endorse the method of designating colors (known as the ISCC-NBS method) developed by the Inter-Society Color Council at the National Bureau of Standards, and shall print this endorsement in its annual report and in the compilation of A.S.T.M. Standards on Textile Materials.

Committee D-13 has adopted this year a Recommended Practice for Calculating the Number of Tests to be Specified in Determining an Average Characteristic of a Textile Material. The procedure was developed by Subcommittee B-5 on Sampling, Presentation, and Interpretation of Data (A. G. Ashcroft, chairman). Committee D-13 is thus the first standing committee to provide its subcommittee chairmen with a method, based on the statistical theory of probability for determining the proper number of tests, and to officially recommend its use in the development of textile standards. The procedure has already been put into use by several subcommittees.

The present membership of Committee D-13 is 211; of whom 96 are classified as producers, 46 as consumers, and 69 as general interest members.

Recommendations Affecting Standards

Committee D-13 is presenting for publication as tentative one new specification and eight new methods of test for textile materials, and is recommending the revision of four tentative standards, the revision of three existing standards for immediate adoption, the tentative revision of six existing standards, the adoption as standard of five tentative standards and of tentative revisions of two existing standards, and the withdrawal of a tentative revision of an existing standard. The revisions being presented are included in detail in the appendix and are accompanied, where necessary, by brief statements of the reasons for such revisions. The new tentative standards and existing standards affected are listed in Table I and are

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Spec. for All Wool, All Cotton, and Wool and Cotton Blanketing (Household)	34	2	59
Testing Woven Asbestos Cloth	28	0	67
Testing and Tolerances for Glass Yarn	29	0	66
Testing and Tolerances for Woven Glass Tapes	27	0	68
Testing and Tolerances for Woven Glass Fabrics	28	0	67
Testing and Tolerances for Woven Glass Tubular Sleeving and Braids	25	0	70
Test for Resistance of Textile Fabrics to Water	44	0	51
Test for Resistance of Textile Fabrics and Yarns to Moths	37	0	58
Test for Shrinkage of Grease Wool (Laboratory Scale Operations)	35	0	60
II. Revisions of Tentative Standards			
Spec. for Bleached Wide Cotton Sheeting (D 503 - 38 T)	43	2	50
Spec. and Test for Fineness of Wool Tops (D 472 - 37 T)	35	0	60
Test for Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide (D 539 - 39 T)	35	0	60
Definitions of Terms Relating to Textile Materials (D 123 - 39 T)	67	0	28
III. Revisions of Standards			
Testing and Tolerances for Cotton Yarns and Thread (D 180 - 37), immediate adoption	58	0	37
Testing and Tolerances for Hose Ducks and Belt Ducks (D 181 - 36), immediate adoption	42	0	53
Testing Pile Floor Covering (D 418 - 39), immediate adoption	40	0	55
Spec. for Textile Testing Machines (D 76 - 39)	60	0	35
Spec. and Test for Asbestos Roving for Electrical Purposes (D 375 - 37)	28	0	67
Spec. and Test for Asbestos Yarns (D 299 - 37)	29	0	66
Spec. and Test for Asbestos Tape for Electrical Purposes (D 315 - 37)	28	0	67
Testing and Tolerances for Cotton Sewing Threads (D 204 - 39)	47	0	48
Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 - 38)	36	1	58
IV. Adoption of Tentative Standards as Standard			
Spec. for Terry (Turkish) Toweling (D 505 - 38 T)	32	2	61
Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 39 T)	38	0	57
Test for Fiber Length of Wool (D 519 - 38 T)	37	0	58
Testing Wool Felt (D 461 - 38 T), as revised	35	0	60
Definitions of Terms Relating to Textile Materials (D 123 - 39 T)	73	0	22
V. Adoption as Standard of Tentative Revisions of Standards			
Spec. for Textile Testing Machines (D 76 - 39), as revised	55	0	40
Spec. and Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 - 34), as revised	45	0	50
VI. Withdrawal of Tentative Revision of Standard			
Testing Woven Textile Fabrics (D 39 - 39; ASA L5 - 1939)	55	0	40

also referred to under the subcommittee activities.

These recommendations have been submitted to letter ballot of the committee which consists of 211 members, the results being shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

The recommendations being submitted indicate that the various subcommittees and sections have been quite active during the past year. Many important projects are under consideration and are mentioned briefly as follows:

Subcommittee A-1 on Cotton and Its Products (K. B. Cook, chairman):

Section I on Cotton (R. W. Webb, chairman) has developed two methods, one on cross-sectioning and one on neppiness, which are included in the extensive revision[1] to be recommended in the Tentative General Methods of Testing Cotton Fibers (D 414 – 37 T).[2]

The agenda of the section includes studies of the flat-bundle test for strength, polarized light method for maturity, and photoelectric methods for length of fibers.

Section II on Cotton Yarns and Threads (A. C. Clifford, chairman) is recommending for immediate adoption a revision and rearrangement,[3] of the Standard General Methods of Testing and Tolerances for Cotton Yarns and Threads (D 180 – 37). A revision for publication as tentative in the Standard Methods of Testing and Tolerances for Cotton Sewing Threads (D 204 – 39) is also recommended, as given in the Appendix.

A tentative revision of Methods D 180 covering a test for appearance has been published for two years, during which time the number of users has materially increased. The section feels that additional experience is desirable and, therefore, recommends that the revision be continued as tentative.

Statistical analyses are in progress to develop requirements for number of tests in Standard Methods D 180 and D 204.

Studies are being conducted on the tension necessary to straighten a yarn without stretching it, and the tolerances for cotton yarn numbers.

Section III on Light and Medium Cotton Woven Fabrics (R. T. Fisher, chairman) is recommending the adoption as standard of the tentative revision in the Standard Specifications and Methods of Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 – 34), amended as proposed in the Appendix.

Section IV on Tire Fabrics (Martin Castricum, chairman) is conducting a study of a testing procedure to evaluate the relative merits of the bone-dry and conditioned methods.

The section is recommending a further tentative revision, as given in the Appendix, of the Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 38).

Section V on Heavy Cotton Woven Fabrics (B. L. Whittier, chairman) is recommending for immediate adoption a revision, as given in the Appendix, of the Standard Methods of Testing and Tolerances for Hose Ducks and Belt Ducks (D 181 – 36), covering the deletion of certain test methods and the substitution of references to the Standard General Methods of Testing Woven Textile Fabrics (D 39).

The section is studying moisture regain of heavy woven fabrics under standard atmospheric conditions. A new approach is being made to the old problem of jaw breaks in testing heavy woven fabrics.

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[1] See Editorial Note, p 412.

[2] 1939 Book of A.S.T.M. Standards, Part III, p. 987.

[3] These revised methods were adopted as standard by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 156.

Subcommittee A-2 on Rayon and Its Products (A. M. Tenney, chairman):

Section I on Rayon (A. M. Tenney, chairman) recommends the adoption as standard of the Tentative Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 39 T).[4] The principal problem before the section is the development of a method for determining the commercial weight of rayon yarn.

Section II on Rayon Fabrics (Alexis Sommaripa, chairman) recommends that the Tentative Method of Test for Resistance to Yarn Slippage in Silk, Rayon, and Silk-Rayon Woven Fabrics (D 434 - 36 T) be continued as tentative, pending the results of its study of means to control shrinkage which has an important bearing on the results of the test for slippage.

Section III on Rayon Staple and Spun Rayon Yarn (K. B. Cook, chairman) is collaborating with Section I in the development of a method for determining the commercial weight of rayon yarns, and with Section I of Subcommittee B-1 in the development of a method for determining the resiliency of rayon staple fiber.

Subcommittee A-3 on Wool and Its Products (A. G. Ashcroft, chairman):

Section I on Wool (Werner von Bergen, chairman) recommends for publication as tentative a Method of Test for Shrinkage of Grease Wool (Laboratory Scale Operations).[5] This method has been published as information for the past year and has proven satisfactory.

The Tentative Method of Test for Fiber Length of Wool (D 519 - 38 T)[6] has been published as tentative for two years; no criticism of the method has been received during that time and the section therefore recommends that it be adopted as standard.

The amended U. S. Government Requirements for Wool Tops were issued January 1, 1940, and the section is recommending that they be substituted for the requirements appearing in the Tentative Specifications and Methods of Test for Fineness of Wool Tops (D 472 - 37 T), as given in the Appendix, and that the specifications as revised be continued as tentative. Members of the section cooperated in the development of these requirements through the submission of 86,000 measurements on 26 samples.

Members of the section have also been the chief cooperators in the development of methods used in the Wool Scouring Laboratory, recently established in Boston by the U. S. Treasury Department for testing raw wool samples for determining duties on imports.

The agenda of the section includes: (*1*) the development of a method for the determination of moisture in wool tops for the purpose of specifying the limits of variation from the standard; (*2*) statistical analysis of measurements of wool tops with a view to defining the grades of this material in purely mathematical terms; (*3*) length specifications for wool top as used by the Wool Top Exchange; and (*4*) a method of tension testing of wool top, and woolen and worsted roving.

Section II on Felt (G. H. Harnden, chairman) is recommending the adoption as standard of the Tentative Methods of Testing Wool Felt (D 461 - 38 T), revised as given in the Appendix.

Section III on Woolen and Worsted Yarns (Fred Noechel, chairman) is making statistical analyses to determine the number of tests to be made for yarn number and breaking strength of woolen and worsted yarns.

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[4] 1939 Book of A.S.T.M. Standards, Part III, p. 1007.

[5] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 485.

[6] 1939 Book of A.S.T.M. Standards, Part III, p. 1041.

Section IV on Pile Floor Coverings (J. N. Dow, chairman) is recommending for immediate adoption certain revisions of the Standard Methods of Testing Pile Floor Covering (D 418 – 39) as given in the Appendix.

Progress is being made with the problem of standardizing machine wear tests and service tests, in which the National Bureau of Standards is collaborating.

Specifications for a machine to be tried out for rating "shedding" properties have been distributed to the members of the section.

The section is working on the development of soiling and crushing tests, and a method of rating color fastness. Statistical studies are being made to determine the precision of analysis of Axminster carpet.

Section V on Woolen and Worsted Fabrics (F. A. Prisley, chairman) recommends that the Tentative Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 – 37 T), which have been tentative for three years without revision, be continued as tentative, since some objections to the methods have been received and certain pending legislation may involve some changes. Work on necessary revisions is in progress, which includes an interlaboratory check on the method for the determination of wool fiber content.

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman):

This subcommittee is recommending for publication as tentative, certain revisions of the Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 – 37), for Asbestos Yarns (D 299 – 37), and for Asbestos Tape for Electrical Purposes (D 315 – 37), as given in the Appendix.

New Methods of Testing Woven Asbestos Cloth are proposed for publication as tentative.[7]

Subcommittee A-4 is continuing its investigations of methods and instruments for thickness measurements, the effect of heat on various grades of asbestos cloth, and iron in asbestos. The development of methods of testing asbestos braided sleeving is in progress.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (A. R. Howe, chairman):

This subcommittee is working on necessary revisions of the Tentative Methods of Testing and Tolerances for Single Jute Yarn (D 541 – 39 T) and the Standard Specifications and Methods of Test for Cuban (Jute) Raw Sugar Bags (D 275 – 33).

Studies are being made of comparative results obtained by the pendulum and constant specimen-rate-of-load types of testing machines. Statistical methods are being applied to determine the precision of the test methods.

Subcommittee A-6 on Household and Garment Fabrics (Ruth O'Brien, chairman):

The subcommittee recommends that the Specifications for All Wool, All Cotton, and Wool and Cotton Blanketing (Household) be accepted for publication as tentative.[8] Owing to the wide variety of blanketing constructions, these specifications answer the need for a classification based on minimum physical requirements which can be used as a buying and selling guide. The classification and physical requirements are based on the analyses of a large number of commercial blanket fabrics.

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[7] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 434.

[8] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 436.

A revision, as given in the Appendix, is recommended in the classification of sheetings appearing in the Tentative Specifications for Bleached Wide Cotton Sheeting (D 503 – 38 T).

No criticisms have been received of the Tentative Specifications for Terry (Turkish) Toweling (D 505 – 38 T)[9] and accordingly the subcommittee recommends that they be adopted as standard.

The Tentative Specifications for Single-Ply Bleached Cotton Broadcloth (D 504 – 38 T) have been published as tentative for two years; however, in view of the work in progress on the development of a method for the determination of the degree of mercerization, the subcommittee recommends that these specifications be continued as tentative.

The subcommittee has in progress the preparation of specifications for corduroy, dish toweling, and upholstery fabrics, and the development of a method of testing blanketing for heat transmission.

Subcommittee A-8 on Glass Fiber and Its Products (Dean Harvey, chairman):

This subcommittee recommends for publication new Tentative Methods of Testing and Tolerances for Glass Yarn, for Woven Glass Tapes, for Woven Glass Fabrics, and for Woven Glass Tubular Sleeving and Braids.[10] These proposed tentative standards supply a need for test methods and tolerances for the new textile products being made of glass fiber.

The subcommittee is making a comparative study of the results obtained in breaking tests of glass yarn by the pendulum and constant specimen-rate-of-load types of testing machines.

Section III on Fabrics (K. N. Mathes, chairman) is working on abrasion tests for glass yarns and fabrics, and is also participating in the work of a joint committee, composed of representatives of cotton, asbestos, and glass tape interests, on the study of methods of measuring thickness.

Subcommittee B-1 on Methods and Machines (W. D. Appel, chairman):

Section I on Methods (W. D. Appel, chairman) recommends that the Tentative Methods for Identification of Fibers in Textiles and Quantitative Analysis of Textiles (D 276 – 37 T) be continued as tentative in view of the fact that a complete revision of the methods is now in progress. The revision will probably result in the division of these methods into two separate tentative standards, one covering identification methods and the other on methods of quantitative analysis.

The section is recommending an editorial revision of the Standard General Methods of Testing Woven Textile Fabrics (D 39 – 39), as given in the Appendix. It also recommends the withdrawal of the revision of Methods D 39 covering the raveled strip method which was published as tentative in 1937.[11] The withdrawal is based on pertinent objections to the limit placed on the applicability of the method.

Other work in progress consists of: (*1*) the development of an accelerated aging test for textiles; (*2*) tests for resiliency of raw fibers; (*3*) tests for resistance of fibers and fabrics to soiling; and (*4*) the preparation of a table of basic properties of textile fibers.

Section II on Machines (H. J. Ball, chairman) recommends that the revision published as tentative in 1938 of the Standard Specifications for Textile Testing Machines (D 76 – 39), which provides specifications for bursting testers be further revised, as given in the Appendix, and approved for adoption as standard. This section is also recommending a tentative revision of Specifications D 76 to provide a method of calibrating thickness gages, as set forth in the Appendix.

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[9] 1939 Book of A.S.T.M. Standards, Part III, p. 980.

[10] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 467, 476, 471, and 474, respectively.

[11] 1939 Book of A.S.T.M. Standards, Part III, p. 1142.

The study of the effect of rate-of-loading on the strength of yarn as determined on type B testing machines is being continued. Also, work is in progress on the preparation of specifications for twist testers.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman):

This subcommittee recommends the adoption as standard of the definitions included in the present Tentative Definitions of Terms Relating to Textile Materials (D 123 – 39 T),[12] with the exception of the definitions for standard moisture regain and crimp which are being retained as tentative. Also, it is recommended that definitions of the following terms be accepted for publication as tentative:[13] carrier, standard condition, fineness, textile glass, length distribution, maturity, moisture equilibrium, sliver, and twine.

A glossary of terms relating to textile materials is published in the Compilation of A.S.T.M. Standards on Textile Materials and the subcommittee is recommending the addition to the glossary of definitions and illustrations of certain common defects in fabrics.

The subcommittee has a number of definitions under consideration. It is also working on a standardized form of expressing yarn construction.

Subcommittee B-4 on Bleaching, Dyeing, and Finishing (W. M. Scott, chairman):

This subcommittee is recommending for publication two new methods of test, namely, a Tentative Method of Test for Resistance of Textile Fabrics to Water, and a Tentative Method of Test for Resistance of Textile Fabrics and Yarns to Moths.[14] It also recommends certain revisions of the Tentative Methods of Test for Fluidity of Cellulose Fibers (D 539 – 39 T), as given in detail in the Appendix.

In view of a further study being given to the Tentative Method of Test for Volumetric Determination of Small Amounts of Copper in Textiles (D 463 – 37 T), the subcommittee recommends that this method be continued as tentative.

Subcommittee B-4 has on its agenda the development of: (*1*) qualitative and quantitative methods of test for the determination of synthetic resin finishes on textiles; (*2*) a method for measuring the acidity and alkalinity of textile fabrics; and (*3*) a method for evaluating so-called "bacteria-static" finishes on textiles.

Subcommittee B-5 on Sampling, Presentation, and Interpretation of Data (A. G. Ashcroft, chairman):

The work of this subcommittee is quite different from that of the other subcommittees in that it cooperates with the other subcommittees in making statistical studies of data with a view to establishing the number of tests required to give a desired precision. The subcommittee has been very active, and as indicated elsewhere in this report, a number of the subcommittees are conducting such studies. For the purpose of guiding this work, Subcommittee B-5 has prepared a Proposed Recommended Practice for Calculating the Number of Tests to be Specified in Determining an Average Characteristic of a Textile Material. It is planned to publish this proposed recommended practice, as information, if approved by letter ballot of the committee, in the 1940 Compilation of A.S.T.M. Standards on Textile Materials.

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[12] 1939 Book of A.S.T.M. Standards, Part III, p. 970.

[13] These definitions were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 428.

[14] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 424 and 422, respectively.

Subcommittee B-6 on Papers (R. H. Brown, chairman):

This subcommittee arranged for the presentation of the following papers at the March, 1940, meeting: (No papers session was held at the October, 1939, meeting.)

"The Use of Synthetic Organic Chemicals in Textile Finishes," by D. E. Truax,

"Some Observations and Preliminary Conclusions Concerning the Influence of Variety, Locality of Growth, and Season on the Fiber and Spinning Properties of Cotton, with Special Reference to Two Selected Varieties for the Crop Years of 1936 and 1937," by R. W. Webb,

"Standardization of Color Names—The ISCC-NBS Method," by Dorothy Nickerson,

"Quality Criterions for Spun Rayon Fabrics," by Alexis Sommaripa, and

"Committee D-13 and the A.S.T.M. Standards on Textile Materials," by H. J. Ball.

Abstracts of the first three papers will appear in the 1940 edition of the compilation of A.S.T.M. Standards on Textile Materials.

Special Committee for Testing of Textile Finishes (L. B. Arnold, Jr., chairman):

Edwin C. Dreby was appointed research associate and began work on this industrially sponsored investigation on August 1, 1939, at the National Bureau of Standards. The object of the project is the development of methods and instruments for the physical evaluation of the "hand" of fabrics, thereby making it possible to measure the effect of various finishing agents upon this important, but at present indeterminate, property of textile materials. Analysis of "hand" has yielded a measure of the extent of correlation between the sensory evaluation of closely similar fabrics by experts and the terms used to describe their evaluation of the property. The analysis has indicated also what degrees of difference can be detected by the experts between individual fabrics and has thus established a basis for determining the limits of precision for instruments. Work is now being concentrated upon the evaluation of three factors best described by the terms limpness, smoothness, and fullness. Excellent correlation has been established between limpness measurements obtained with the Schiefer flexometer, by the Pierce hanging-heart loop method, and the expert's evaluation of the same property. Simple devices and methods for measuring surface smoothness are being developed and studied.

The interest of the Special Committee in methods for evaluating the fire resistance of fabrics is probably covered by the "Recommended Requirements for Flameproofing of Textiles" developed at the National Bureau of Standards and adopted by the National Fire Protection Association as a tentative standard. A copy has been transmitted to Subcommittee B-4 on Bleaching, Dyeing, and Finishing for its consideration.

Acknowledgment is made of the close cooperation and valuable assistance and advice which have been given by the National Bureau of Standards and its staff. One change has occurred in the personnel of the Directing Committee. Laura E. Pratt has been appointed to take the place of Elizabeth Weirick, resigned. The great majority of the present subscribers to the investigation have agreed to continue their support for another year, and additional sponsors have been obtained.

At the March, 1940, meeting the present officers of the committee were re-elected for the two-year period from June, 1940, to June, 1942.

This report has been submitted to letter ballot of the committee which consists of 211 members; 95 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-13 presented to the Society through Committee E-10 on Standards a revision of the Tentative General Methods of Testing Cotton Fibers (D 414 – 37 T). The tentative methods in their revised form were accepted by Committee E-10 on September 5, 1940, and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 446.

APPENDIX

PROPOSED REVISIONS IN STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentative standards covering textile materials which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the standard appears in its present form.

REVISIONS OF TENTATIVE STANDARDS

Tentative Specifications for Bleached Wide Cotton Sheeting (D 503 - 38 T):[1]

The purpose of the revision is to overcome objection to the use of the name "muscale" which was coined to distinguish one construction.

Section 3.—Change the classification of materials from its present form: namely,

Percale
Muscale
Unnamed
Muslin { Grade *A*
Grade *B*
Grade *C*

to read as follows:

Type 1, percale.
Type 2, fine-count muslin.
Type 3.
Type 4, heavy-weight muslin.
Type 5, medium-weight muslin.
Type 6, light-weight muslin.

Table I.—Change the names of the materials in the headings of this table to the respective type numbers as indicated in the revision of Section 3 above.

Tentative Specifications and Methods of Test for Fineness of Wool Tops (D 472 - 37 T):[2]

The revisions in these specifications and methods are being recommended in order to bring the requirements for wool tops in full agreement with the government standards.

Table I.—Replace the present table of requirements by the accompanying Table I.

Section 4.—Change from its present form to read as follows:

4. Samples comprising two or more sections of sliver, each at least 1 yd. in length, shall be submitted in accordance with the following requirements:

Size of Lot,[a] lb.	Number of Balls Sampled	Number of Samples
5000 and under	2	1
Over 5000 to 20 000	3	1
Over 20 000	3 for each 20 000 lb.	1 for each 20 000 lb.

[a] The term "lot" as used here refers to a single combing lot or portion thereof.

Section 10.—Add a new note to read as follows:

NOTE: *Example.*—If a lot of 5000 lb. of grade 64's wool tops is to be tested, at least 1 yd. of the wool top is taken from each of 2 balls as the sample. The required 600 fibers are taken in equal proportions from the two lengths of sliver, or 300 fibers from each. If the lot runs from 5,000 to 20,000 lb., three 1-yd. lengths, one from each of 3 balls, will be required for the sample. In this case, each length would supply 200 fibers or 600 fibers for the three lengths.

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[1] 1939 Book of A.S.T.M. Standards, Part III, p. 977.
[2] *Ibid.*, p. 1035.

Figure 6.—Replace the illustration of the wedge measure by the accompanying Fig. 1.

Tentative Method of Test for Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide (D 539 - 39 T):[3]

Section 2.—Add a footnote to refer to the viscosimeter described in Paragraph (*a*) to read as follows:

> Suitable viscosity apparatus, originally designed by the Technical Association of the Pulp and Paper Industry for determining the cuprammonium viscosity of pulp, may be obtained from manufacturers of scientific apparatus. Calibrated viscosimeters may be obtained from the Shirley Institute, Didsbury, Manchester, England.

In Paragraph (*d*) omit "when $d = 1$ and $F = 100$" which follows Eq. 2, and change Eq. 3 to read as follows:

$$K = \frac{dFt^2 - Ct}{dF} \qquad (3)$$

Change the paragraphs describing constants C and K to read as follows:

> *Constant C* shall be determined first. A liquid of known low fluidity and for which the kinetic energy of flow (constant K) is sufficiently small to be negligible shall be used. A suitable liquid for this purpose is an oil (Note) having a known density and a calibrated fluidity between 3 and 5 rhes at a temperature of 20 C. The calibrating liquid shall be placed in the viscosimeter and maintained while measurements are being made at a temperature within plus or minus 0.5 C. of the temperature at which its fluidity is known. The time of flow t shall be determined in accordance with Section 6 (*c*). The constant C shall be calculated from

TABLE I.—REQUIREMENTS FOR WOOL TOPS.*

Grade	80's	70's	64's	62's	60's	58's	56's	50's
Fineness Range, microns:								
Min.	18.1	19.6	21.1	22.6	24.1	25.6	27.1	29.1
Max.	19.5	21.0	22.5	24.0	25.5	27.0	29.0	31.5
Fibers, per cent:								
10 to 20 microns, incl., min.	60	50	36	27	18	16	9	4
10 to 25 microns, incl., min.	92	84	....	....	....	....	....	....
10 to 30 microns, incl., min.	....	....	94	88	83	74	64	45
25.1 to 30 microns, incl., max.	8	....	....	....	....	....	....	....
25.1 to 40 microns, incl., max.	....	16	....	....	....	....	....	....
30.1 to 40 microns, incl., max.	0.25 (0.5)[a]	2	6	12	17	....	....	....
30.1 to 50 microns, incl., max.	....	....	....	....	....	26	36	55
40.1 to 50 microns, incl., max.	....	0.25 (0.5)[a]	0.33 (1)[a]	0.50 (1)[a]	0.50 (1)[a]	2	5	10
50.1 and over microns, max.	....	....	....	....	....	0.75 (1.5)[a]	1 (2)[a]	1.25 (2.5)[a]
Minimum number of fibers required for test, per sample	400	400	600	600	800	800	1200	1200

* These requirements conform to the U. S. Government Requirements for Wool Tops promulgated January 1, 1940.

[a] Numbers in parentheses represent maximum percentage of fibers of that range permissible in substandard grades.

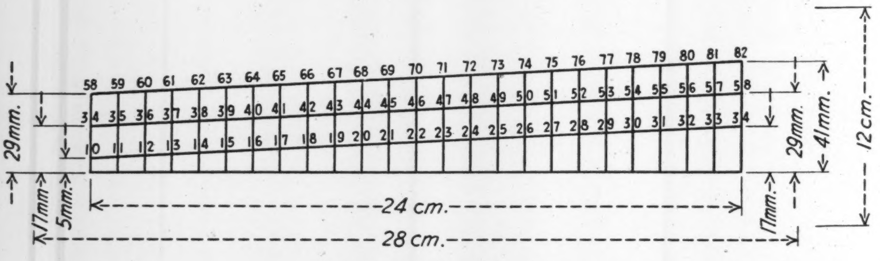

FIG. 1.—Wedge Measure.

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[3] 1939 Book of A.S.T.M. Standards, Part III, p. 981.

Eq. 2 by assuming that K is equal to zero and substituting the known values of d and F, and the determined value of t.

Constant K shall be determined under the same temperature conditions and by the same procedure as used for the determination of constant C except that an oil (Note) having a known density and a calibrated fluidity between 46 and 48 rhes shall be used as the calibrating liquid. The constant K shall be calculated from Eq. 3 by substituting the known values of d, F, C, and the determined value of t.

NOTE.—Calibration oils in the specified ranges of fluidity can be obtained from the National Bureau of Standards, Washington, D. C. Aqueous solutions of glycerol may be substituted for the calibrated oils. The first solution shall have a density of 1.1681 and a fluidity of 6.83 rhes at a temperature of 20 C. and the second solution shall have a known density and an exactly determined fluidity of about 50 rhes.

Section 3.—Add a new paragraph to the note following this section, with appropriate references in Section 3, to read as follows:

Published data indicate that with 0.5 per cent cellulose solutions of low fluidity, a variation in the copper concentration of 0.5 g. per liter will cause a change of 0.01 rhe in the fluidity, and a variation in the ammonia (NH_3) concentration of 20 g. per liter will cause a change of 0.4 to 0.6 rhe in the fluidity. Data is not available for cellulose solutions with fluidities higher than 18 rhes. For routine work the above accuracy should be sufficient, but if greater accuracy is desired, the permissible variations in the cuprammonium solution may be reduced to plus or minus 0.1 g. of copper and plus or minus 5 g. of ammonia (NH_3) per liter.

REVISIONS OF STANDARDS FOR IMMEDIATE ADOPTION

Standard Methods of Testing and Tolerances for Hose Ducks and Belt Ducks (D 181 - 36):[4]

Section 4.—Change to read as follows by the addition of the italicized words and the omission of those in brackets.

4. All test procedures, [not covered in the following Sections 5 to 10] *except as specified in Section 5,* shall be in accordance with the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.

Sections 5 to 10.—Omit Sections 5, 7, 8, 9, and 10 on width, count, thickness, crimp, and breaking strength, respectively, and renumber Section 6 as Section 5.

Standard Methods of Testing Pile Floor Covering (D 418 - 39):[5]

Section 2.—Omit Paragraph (*a*) which reads as follows:

(*a*) *Unconditioned Weight.*—The weight of the fabric when samples for weight determination are cut from it is the unconditioned weight. A specification of the weight of a fabric must also specify the moisture condition.

Section 14.—Add a new sentence at the end of this section to read as follows:

The minimum area of a dissection sample shall be 16 sq. in. with a minimum linear dimension of 2 in.

Section 15.—Add a new Paragraph (*a*) to read as follows, relettering the subsequent paragraphs accordingly:

(*a*) *Total Weight.*—The weight of the sample before dissection shall be the total conditioned weight of the fabric.

NOTE.—In the case of certain special fabrics which may contain considerable nonfibrous material as coating, it may be desirable first to remove such material by a suitable method before weighing. In such a case the weight obtained is the total yarn weight of the fabric.

Reletter Paragraph (*c*) as (*d*) and change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*d*) Clean Pile Yarn.—The average of the weights of the samples of pile yarn, after [extraction to remove fats and after desizing to remove sizing materials] *desizing and removal of fats in accordance with Section 3 of the Tentative Methods for Identification of Fibers in Textiles and Quantitative Analysis of Textiles (A.S.T.M. Designation: D 276 - 37 T) of the American Society for Testing Materials,*[6] shall be the conditioned weight of clean pile yarn.

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[4] 1939 Book of A.S.T.M. Standards, Part III, p. 421.
[5] *Ibid.*, p. 473.
[6] *Ibid.*, p. 951.

Note.—When it is desired to obtain a fiber analysis of the pile yarn the procedures given in Tentative Methods D 276[6] shall be followed.

Reletter Paragraph (*h*) as (*i*), omit the note following it, and change the paragraph to read as follows by the addition of the italicized words and the omission of those in brackets:

(*i*) Clean Backing Yarns.—When required, the average of the weights of the samples of warp yarn, stuffer yarn, and filling yarn after [extractions to remove fats and after desizing to remove size] *desizing and removal of fats in accordance with Section 3 of Tentative Methods D 276*,[6] shall be the conditioned weight of clean warp yarn, clean stuffer yarn, and clean filling yarn, respectively.

New Sections.—Add as new Sections 16 and 17 the descriptions of procedures for measuring tuft length and determining precision of results as appended hereto,[7] renumbering the subsequent sections accordingly.

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Textile Testing Machines (D 76 - 39):[8]

The committee recommends the following tentative revision of this standard in order to provide for a calibration method for thickness gages:

Section 8.—Add a new Paragraph (*c*) on calibration of thickness gages to read as follows:

(*c*) *Calibration.*—The gage shall be calibrated for the actual load exerted by the presser foot by means of any device so arranged as to measure the total vertical force exerted by the presser foot at the several gage readings or presser foot levels selected for calibration. The presser foot shall be brought to each calibration level from a higher one.

NOTE.—It is suggested that the calibration may be accomplished by supporting the presser foot on a horizontal strap suspended from a weighing device so arranged that it may be raised or lowered gradually and leveled at any required position.

Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 - 37):[9]

The committee recommends the following tentative revision of this standard in order to provide for standard test conditions:

New Section.—Under methods of testing add the following as a new Section 7 on test conditions, renumbering the subsequent sections accordingly:

7. *Test Conditions.*—All specimens shall be conditioned to moisture equilibrium in a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

NOTE.—For purpose of tests, it is recognized that samples reach moisture equilibrium after exposure for at least 4 hr., unless otherwise specified, under the above conditions.

Standard Specifications and Methods of Test for Asbestos Yarns (D 299 - 37):[10]

The committee recommends the following tentative revision of this standard in order to standardize the statement on test conditions and to provide needed methods for the determination of twist:

Section 2.—Add a new Paragraph (*e*) to read as follows, relettering the subsequent paragraphs accordingly:

(*e*) *Direction of Twist.*—The direction of twist shall be as defined in the Tentative Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123) of the American Society for Testing Materials, as follows:

Twist, Direction of.—A yarn or cord has S twist if, when held in a vertical position, the spirals conform in direction of slope to the central portion of the letter "S," and Z twist if the spirals conform in direction of slope to the central portion of the letter "Z."

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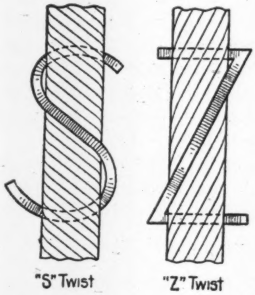
[7] This revision was adopted as standard by the Society and has been incorporated in the methods which appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 180.

[8] 1939 Book of A.S.T.M. Standards, Part III, p. 364.

[9] 1939 Book of A.S.T.M. Standards, Part III, p. 385.

[10] *Ibid.*, p. 388.



New Section.—Add as a new Section 7 on twist to read as follows, renumbering the subsequent sections accordingly:

7. The directions and turns per inch of twist shall be mutually agreed upon by the manufacturer and the purchaser.

Section 7.—Change this section to read as follows by the addition of the italicized words and the omission of those in brackets, renumbering as Section 8:

8. [When it is specified that test specimens shall be conditioned, it is understood to mean exposure for the specified time to an] *All specimens shall be conditioned to moisture equilibrium in a standard* atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus *or minus* 2 F. (1.1 C.) in temperature.

Note.—For purpose of tests, it is recognized that specimens reach moisture equilibrium after exposure for at least 4 hr., unless otherwise specified, under the above conditions.

New Section.—Under methods of testing, add a new Section 13 on determination of twist to read as follows:

13. *Twist*: (*a*) *Direction of Twist.*—The direction of twist shall be determined in accordance with Section 2 (*e*).

(*b*) *Turns in Single Yarn.*—The turns per inch of twist in single yarn shall be determined on any standard single yarn twist tester. The first 2 yd. of any package of yarn shall be discarded and all possible care shall be exercised in handling samples to prevent a change in the existing twist. The yarn, drawn just taut enough to prevent sagging, shall be gripped in the twist counter with clamps set 4 in. apart. One clamp shall be rotated until the twist is removed as indicated by the yarn breaking under its own weight or by visual examination of the sample. The twist of yarn in a shipment shall be the average of four tests from each of ten tubes or bobbins.

(*c*) *Turns in Plied Yarn.*—The turns per inch of twist in plied yarn shall be determined as described in Paragraph (*b*) with the following exceptions: The clamps shall be set 10 in. apart and a tension equal to the weight of 100 yd. of yarn shall be applied.

Standard Specifications and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 - 37):[11]

The committee recommends the following tentative revision of this standard in order to provide for standard test conditions:

New Section.—Under methods of testing, add a new Section 15 on test condition to read as follows, renumbering the subsequent sections accordingly:

15. All specimens shall be conditioned to moisture equilibrium in a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

NOTE.—For purpose of tests, it is recognized that samples reach moisture equilibrium after exposure for at least 4 hr., unless otherwise specified, under the above conditions.

Standard Methods of Testing and Tolerances for Cotton Sewing Threads (D 204 - 39):[12]

Section 5.—Change this section to read as follows by the addition of the italicized figure and the omission of that in brackets:

5. The number of yards per package or per pound shall be not less than [96] *95* per cent of the number of yards per package or per pound specified.

Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 - 38):[13]

The following tentative revision of these methods is proposed in order to broaden the scope to include rayon cord and rayon cord fabrics, and to provide for a sampling procedure:

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[11] 1939 Book of A.S.T.M. Standards, Part III, p. 391.
[12] *Ibid.*, p. 418.
[13] *Ibid.*, p. 431.

Section 1.—Change to read as follows by the addition of the italicized words and the omission of the word in brackets:

1. These methods of testing and tolerances apply to woven cotton cord fabric, *woven rayon cord fabric,* [and] cotton cord on cones, *and rayon cord on cones,* specifically designed for use in the manufacture of pneumatic tires.

New Section.—Add a new Section 11 on sampling procedure, renumbering the subsequent sections accordingly:

11. *Sampling.*—A minimum of 25 yd. shall be drawn from the outside of the cone before taking the sample. The sample shall be so handled that there will be no change in twist from the time the sample is drawn until the test is started. In each determination only one observation shall be made on an individual end, whether in the form of woven cord fabric or cord on cones.

ADOPTION OF TENTATIVE STANDARD AS STANDARD

Tentative Methods of Testing Wool Felt (D 461 - 38 T):[14]

Section 12.—Add a new Note 3 to this section to read as follows:

NOTE: *Precautions.*—Care should be taken to insure that all loose fibers are removed from the filter papers, beakers, and extraction thimbles used during the analysis.

ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Textile Testing Machines (D 76 - 39):[8]

The new Sections 9 and 10 on bursting testers published as tentative[15] for two years without criticism are now recommended for adoption as standard, revised as follows:

Section 9.—Change this section from its present form to read as follows:

9. *Attachment for Type A Machines.*—The attachment for making the bursting test on a type A machine shall be of such design that the specimen shall be held securely by a ring-clamp mechanism of 1.750 ± 0.001 in. in internal diameter, and that the center of the specimen shall be pressed against a polished steel ball 1.000 ± 0.001 in. in diameter until a burst is produced. The direction of motion of the ring-clamp shall be at right angles to the initial plane of the specimen. The capacity of the machine shall conform to the requirements of Section 3 (*b*).

Section 10.—Change the second sentence of Paragraph (*c*) from its present form to read as follows:

The ring-clamp shall have a circular aperture 1.200 ± 0.001 in. in diameter.

Standard Specifications and Methods of Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 - 34):[16]

With the following modifications the committee recommends the adoption of the tentative revision[17] of this standard which has been published as tentative for two years:

Section 5.—Change this section to read as follows by the addition of the italicized figures and the omission of those in brackets:

5. As little sizing shall be used in the fabric as is necessary for its proper manufacture. Assuming an average content of natural oils and waxes indigent to the cotton of [3.2] *4.25* per cent, the total percentage of sizing and natural oils and waxes shall not exceed that given below for each type of fabric:

Sheetings	[8.5]	*10.75*	per cent
Drills	[11]	*12.0*	per cent
Sateens	[10.25]	*12.0*	per cent
Broken Twills	[8.15]	*12.0*	per cent

Sizing Test.—The committee recommends the adoption as standard of the test for sizing described in the proposed revised Section 15 with the omission of the words "Preferred Method" from the heading, and the withdrawal of the "alternate method" in the proposed revised Section 16.

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[14] 1939 Book of A.S.T.M. Standards, Part III, p. 1044.
[15] *Ibid.*, p. 1141.
[16] *Ibid.*, p. 395.
[17] *Ibid.*, p. 1142.

EDITORIAL CHANGES IN STANDARDS

Standard General Methods of Testing Woven Textile Fabrics (D 39 - 39):[18]

Sections 9 to 12.—In connection with the description in these sections of the procedures for determining the breaking strength of fabrics add as Figs. 1 and 2 the diagrams illustrating the proper method of placing the specimen in the clamps as shown in the accompanying Figs. 2 and 3.

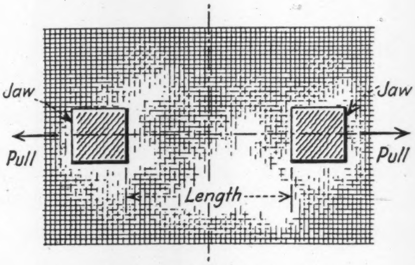

FIG. 2.—Illustration of Grab Test.

Standard Methods of Testing and Tolerances for Cotton Sewing Threads (D 204 - 39):[12]

Section 10.—Change to read as follows by the omission of the figure in brackets and the addition of the note:

10. The breaking strength shall be determined in accordance with Sections 7 and 8, [9] of Standard D 180.

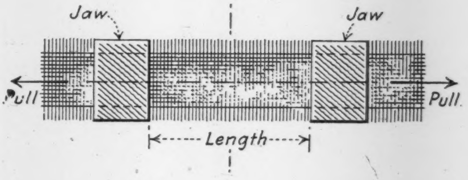

FIG. 3.—Illustration of Strip Test.

NOTE.—If facilities for testing under standard conditions are not available, reference should be made to the method described in the Appendix to Standard D 180.

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[18] 1939 Book of A.S.T.M. Standards, Part III, p. 370.

REPORT OF COMMITTEE D-17
ON
NAVAL STORES

A meeting of Committee D-17 on Naval Stores was held during the annual meeting of the Society in Atlantic City, N. J., on June 26, 1939, at which the program for the ensuing year was discussed.

Subcommittee I on Softening Point of Rosin (C. E. Kinney, chairman) has recommended that the Tentative Method of Test for Softening Point (Ball and Tapered Ring Apparatus) (E 28 – 39 T) be further revised to permit the optional use of a shouldered ring for certain high melting resins, modified rosins, and other products which so contract on cooling that they tend to slip part way out of the tapered ring. This recommendation, together with a drawing of the proposed shouldered ring, has been transmitted to Technical Committee II on Consistency, Plasticity, and Related Properties of Committee E-1 on Methods of Testing for review and consideration. Action has not yet been taken by Committee E-1 on this recommendation. Further collaborative tests on the ball and tapered ring apparatus have been held up pending such action.

The chairman of Subcommittee I is making an investigation of the suitability of the ball and ring method for plant control use. A summary of the final results showing a comparison of the tapered ring and shouldered ring is presented in Table I.

Subcommittee II on Crystallization (E. V. Romaine, chairman) has continued its study of the low-temperature heat-treatment method for determining the crystallizing tendency of rosin. A change in the initial heat treatment was made. Six samples of rosin were sent to each of the members of the subcommittee for test by the modified method. To date only two of the collaborators have reported results.

A proposal to recommend the adoption as standard of the Tentative Methods of Sampling and Grading Rosin (D 509 – 38 T) was submitted to letter ballot of Committee D-17 which consisted of 20 members; 19 members returned their ballots, of whom 8 voted affirmatively, 9 negatively, and 2 members marked their ballots "not voting." It will therefore be necessary for the committee to make a further study of the methods for the purpose of suitably revising them.

On the recommendation of Subcommittee IV on Acid and Saponification Numbers and Unsaponifiable Matter (C. C. Zeigler, chairman), the Tentative Methods of Test for Saponification Number of Rosin (D 464 – 37 T), and Test for Acid Number of Rosin (D 465 – 37 T) are being continued as tentative since a modification of the methods is under consideration in order to provide for the testing of dark colored rosins.

TABLE I.—RESULTS OF SOFTENING POINT DETERMINATIONS ON REFEREE SAMPLES OF RESINS BY COLLABORATING LABORATORIES.

	Limed Wood Rosin		WW Wood Rosin		FF Wood Rosin		Gum Rosin		Gum Rosin		Synthetic Resin	
	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring
F. P. Veitch by W. C. Smith, Naval Stores Research Div., Bureau of Agric. Chemistry and Engr., U. S. Dept. of Agric.												
Individual determinations	136.00 136.50 135.00	137.00 137.00 136.00	79.00 79.50 79.00	79.50 79.50 79.50	73.00 73.00 73.00	73.00 73.00 73.00	76.00 75.50 75.50	76.00 75.50 75.50	71.00 71.00 71.00	71.00 71.00 71.00	152.50 152.25 153.50	153.25 153.00 154.00
Average	136.00	136.50	79.00	79.50	73.00	73.00	75.50	75.50	71.00	71.00	152.75	153.42
Maximum deviation	1.50	1.00	0.50	0.00	0.00	0.00	0.50	0.50	0.00	0.00	1.00	1.00
Maximum deviation from average	1.00	0.50	0.50	0.00	0.00	0.00	0.50	0.50	0.00	0.00	0.75	0.58
Average deviation of shouldered from tapered	+0.50		+0.50		0.00		0.00		0.00		+0.67	
G. A. Hawkins, Southern Pine Chemical Co.												
Individual determinations	136.50 136.50 137.25	137.25 137.50 137.75	80.25 80.25 80.00	80.75 80.50 80.25	73.25 73.25 73.25	73.25 73.50 73.75	75.50 75.75 75.75	75.75 76.00 75.75	70.75 70.75 70.75	71.00 71.00 71.25	152.50 152.50 152.00	153.75 152.50 153.00
Average	136.75	137.50	80.17	80.50	73.25	73.50	75.67	75.83	70.75	71.08	152.33	153.08
Maximum deviation	0.75	0.50	0.25	0.50	0.00	0.50	0.25	0.25	0.00	0.25	0.50	1.25
Maximum deviation from average	0.50	0.25	0.17	0.25	0.00	0.25	0.17	0.17	0.00	0.17	0.33	0.67
Average deviation of shouldered from tapered	+0.75		+0.42		+0.25		+0.16		+0.33		+0.75	
R. J. Sobatzki, The Resinous Products and Chemical Co.												
Individual determinations	134.00 135.50	135.00 136.00	80.80 80.50	81.00 81.00	74.00 73.50	74.00 74.00	75.50 75.50	75.50 75.50	70.70 70.70	70.70 70.50	154.00 154.00 154.50	154.00 154.50 155.00
Average	134.80	135.50	80.70	81.00	73.80	74.00	75.50	75.50	70.70	70.60	154.20	154.50
Maximum deviation	1.50	1.00	0.30	0.00	0.50	0.00	0.00	0.00	0.00	0.20	0.50	1.00
Maximum deviation from average	0.80	0.50	0.20	0.00	0.30	0.00	0.00	0.00	0.00	0.10	0.30	0.50
Average deviation of shouldered from tapered	+0.70		+0.30		+0.20		0.00		−0.10		+0.30	
E. T. Marceau, Hecker Products Corp.												
Individual determinations	134.50 133.75 134.75	134.50 133.75 135.00	79.75 80.50 79.75	80.00 80.75 80.00	73.75 73.25 73.75	73.50 73.25 74.25	75.50 75.25 75.50	75.50 75.50 75.75	71.50 72.00 71.00	73.25 72.25 71.50	154.25 153.25 153.25	155.00 155.50 154.50
Average	134.30	134.40	80.00	80.30	73.60	73.70	75.40	75.60	71.50	72.00	153.60	155.00
Maximum deviation	1.00	1.25	0.75	0.75	0.50	1.00	0.25	0.25	1.00	1.75	1.00	1.00
Maximum deviation from average	0.55	0.65	0.50	0.45	0.35	0.55	0.15	0.15	0.50	1.25	0.65	0.50
Average deviation of shouldered from tapered	+0.10		+0.30		+0.10		+0.20		+0.50		+1.40	
W. A. Kirklin, Hercules Powder Co.												
Individual determinations	136.20 135.60	136.00 135.00	81.20 80.50	81.00 81.00	75.00 74.40	74.80 74.80	76.70 76.00	76.70 76.50	72.20 73.20 72.00	72.50 72.20 73.50	150.50 152.00 153.00 153.50	154.00 154.00
Average	135.90	135.50	80.90	81.00	74.70	74.80	76.40	76.60	72.50	72.70	152.00	154.00
Maximum deviation	0.60	1.00	0.70	0.00	0.60	0.00	0.70	0.20	1.20	1.30	3.00	0.00
Maximum deviation from average	0.30	0.50	0.40	0.00	0.30	0.00	0.40	0.10	0.70	0.80	1.50	0.00
Average deviation of shouldered from tapered	−0.40		+0.10		+0.10		+0.20		+0.20		+2.00	

TABLE I.—*Concluded*

	Limed Wood Rosin		WW Wood Rosin		FF Wood Rosin		Gum Rosin		Gum Rosin		Synthetic Resin	
	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring	Tapered Ring	Shouldered Ring
H. R. Woerner, Armstrong Cork Co.												
Individual determinations	135.75	134.50	81.00	81.00	72.50	73.00	74.75	74.50	73.00	72.50	153.00	153.25
	136.25	135.50	80.50	80.75	73.25	72.75	74.75	75.00	72.50	72.75	153.00	153.50
	136.00	135.00	81.50	81.00	73.00	73.00	75.00	75.00	72.25	71.75	152.25	153.75
Average	136.00	135.00	81.00	80.90	72.90	72.90	74.80	74.80	72.60	72.30	152.80	153.50
Maximum deviation	0.50	1.00	1.00	0.25	0.75	0.25	0.25	0.50	0.75	1.00	0.75	0.50
Maximum deviation from average	0.25	0.50	0.50	0.15	0.40	0.15	0.20	0.30	0.40	0.45	0.55	0.25
Average deviation of shouldered from tapered	−1.00		−0.10		0.00		0.00		−0.30		+0.80	
R. C. Palmer, Newport Industries, Inc.												
Individual determinations	136.00	135.50	79.00	80.50	75.00	74.50	77.80	77.80	72.00	72.00	150.50	152.50
	135.00	136.00	81.00	80.50	75.00	75.50	76.80	77.00	71.50	72.50	153.00	154.00
Average	135.50	135.75	80.00	80.50	75.00	75.00	77.30	77.40	71.75	72.25	151.75	153.25
Maximum deviation	1.00	0.50	2.00	0.00	0.00	1.00	1.00	0.80	0.50	0.50	2.50	1.50
Maximum deviation from average	0.50	0.25	1.00	0.00	0.00	0.50	0.50	0.40	0.25	0.25	1.75	0.75
Average deviation of shouldered from tapered	+0.25		+0.50		0.00		+0.10		+0.50		+1.50	
SUMMARY												
Maximum deviation	1.50	1.25	2.00	0.75	0.75	1.00	1.00	0.80	1.20	1.75	3.00	1.50
Maximum deviation from average	1.00	0.65	1.00	0.50	0.40	0.55	0.50	0.50	0.70	1.25	1.75	0.75
Maximum deviation of shouldered from tapered	+0.75 −1.00		+0.50 −0.10		+0.25 −0.00		+0.20 −0.00		+0.50 −0.10		+2.00 −0.00	
Average deviation of shouldered from tapered	+0.13		+0.20		+0.10		+0.10		+0.16		+1.06	

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 20 members; 17 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. P. VEITCH,
Chairman.

C. F. SPEH,
Secretary.

REPORT OF COMMITTEE D-18

ON

SOILS FOR ENGINEERING PURPOSES

Committee D-18 on Soils for Engineering Purposes has held three meetings during the past year: in Atlantic City, N. J., on June 28, 1939, in Washington, D. C., on December 4, 1939, and in Detroit, Mich., on March 7, 1940.

I. New Tentative Standards

The committee recommends that the following methods of test be accepted for publication as tentative:

Tentative Methods of Test for:

Moisture-Density Relations of Soil-Cement Mixtures,[1]

Wetting-and-Drying Test of Compacted Soil-Cement Mixtures,[1]

Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures,[1] and

Water Absorption and Resistance to Plastic Flow of Soil and Emulsified Asphalt Mixtures.[2]

II. Revision of Tentative Standard

The committee recommends that the Tentative Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 - 35 T)[3] be revised as follows and continued as tentative:

Section 2.—Change from its present form to read as follows:

2. These methods cover procedures for the surveying and sampling, for identification and subgrade profile purposes, of soils for highway subgrades but do not include the examination of soils for their supporting value as foundation materials or stability in slopes.

Note.—Investigation of soils under structures or fills for their supporting value and the stability of slopes in embankments or cuts should not be overlooked by the engineer and where necessary a thorough study of these factors should be made. This will require special procedures and equipment not covered in these methods.

The recommendations appearing in this report have been submitted to letter

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Test for Moisture-Density Relations of Soil-Cement Mixtures[a]	32	6	7
Wetting-and-Drying Test of Compacted Soil-Cement Mixtures[b]	30	5	10
Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures[c]	30	5	10
Test for Water Absorption and Resistance to Plastic Flow of Mixtures of Soil and Emulsified Asphalt[d]	20	9	16
II. Revision of Tentative Standard			
Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 - 35 T)	44	0	1

[a] The classified vote on the proposed Tentative Method of Test for Moisture-Density Relations of Soil-Cement Mixtures was as follows: Affirmative: 6 producers, 16 consumers, 10 general interests; negative: 1 producer, 3 consumers, 2 general interests.

[b] The classified vote on the proposed Tentative Method of Wetting-and-Drying Test of Compacted Soil-Cement Mixtures was as follows: Affirmative: 7 producers, 15 consumers, 8 general interests; negative: 0 producers, 3 consumers, 2 general interests.

[c] The classified vote on the proposed Tentative Method of Freezing-and-Thawing Test of Compacted Soil-Cement Mixtures was as follows: 7 producers, 15 consumers, 8 general interests; negative: 0 producers, 3 consumers, 2 general interests.

[d] The classified vote on the proposed Tentative Method of Test for Water Absorption and Resistance to Plastic Flow of Soil and Emulsified Asphalt Mixtures was as follows: Affirmative: 3 producers, 11 consumers, 6 general interests; negative: 3 producers, 4 consumers, 2 general interests. This recommendation by action of the Society at the annual meeting was referred to Committee E-10 on Standards for consideration, see Summary of Proceedings, p. 22.

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[1] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 294, 297, and 301, respectively.

[2] The recommendation on this proposed tentative method by action of the Society at the annual meeting was referred to Committee E-10 on Standards for consideration, see Summary of Proceedings, p. 22.

[3] 1939 Book of A.S.T.M. Standards, Part II, p. 1048.

ballot of the committee which consists of 70 members; 45 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (W. P. Kimball, chairman) is making good progress in the preparation of a list of definitions.

Subcommittee II on Classification of Soils (E. F. Preece, chairman) will consider at the June meeting the desirability of sponsoring a symposium on classification, to be held at the annual meeting of the Society in 1941.

Subcommittee III on Methods of Sampling Soils (R. R. Litehiser, chairman) has recommended the revision of the Tentative Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 – 35 T), referred to earlier in this report.

Subcommittee IV on Methods of Testing the Physical Characteristics of Soils (C. A. Hogentogler, chairman) is studying the desirability of revising the following methods which are under its jurisdiction:

Standard Methods of:

Preparing Soil Samples for Mechanical Analysis and Determination of Subgrade Soil Constants (D 421 – 39),
Mechanical Analysis of Soils (D 422 – 39),
Test for Liquid Limit of Soils (D 423 – 39),
Test for Plastic Limit and Plasticity Index of Soils (D 424 – 39),
Test for Centrifuge Moisture Equivalent of Soils (D 425 – 39),
Test for Field Moisture Equivalent of Soils (D 426 – 39), and
Test for Shrinkage Factors of Soils (D 427 – 39).

Subcommittee V on Methods of Testing for Stabilized Soils (F. C. Lang, chairman) has proposed for publication as tentative the four methods of test mentioned earlier in this report.

Subcommittee VI on Methods of Testing for Compressibility and Elasticity of Soils (D. M. Burmister, chairman) has concentrated its attention during the year on the consolidation characteristics of soils. A program of research is being carried on to investigate those aspects of the consolidation test about which uncertainties exist, particularly to determine the influence of a number of important factors on the consistency and reliability of results obtained. It is hoped that these data will form the basis for recommendations as to test procedure and test apparatus.

Subcommittee VII on Methods of Testing for Shearing Resistance of Soils (F. J. Converse, chairman) has been sponsoring a cooperative investigation on shear tests in which a number of laboratories are participating.

Subcommittee VIII on Methods of Testing for Mechanical Stability of Soils (L. W. Hamilton, chairman) has been engaged in the development of stabilometer tests and equipment.

Subcommittee IX on Methods of Testing for Bearing Capacity of Soils in Place (Load Tests) (W. S. Housel, chairman) has held several meetings to discuss load tests. Further consideration is required before proposed standard procedures can be logically formulated.

Subcommittee X on Methods of Testing for Bearing Capacity of Piles (Load Tests) (A. E. Cummings, chairman) has gathered for study, data on the various procedures now in use.

Subcommittee XI on Methods of Testing Drainage Properties of Soils (Capillarity and Permeability) (W. J. Schlick, chairman) must await additional research results before undertaking constructive work on its problem.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, C. A. Hogentogler.
Vice-Chairman, W. S. Housel.
Secretary, W. R. Nelson.

This report has been submitted to letter ballot of the committee which consists of 70 members; 40 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. CLEMMER,
Chairman.

W. R. NELSON,
Secretary.

REPORT OF COMMITTEE D-19
ON
WATER FOR INDUSTRIAL USES

Committee D-19 on Water for Industrial Uses and its subcommittees held meetings on June 26 and 27, 1939, during the annual meeting of the Society in Atlantic City, N. J.

A session on water was sponsored by the committee at the 1939 annual meeting, at which a group of five papers[1] were presented and discussed. The committee is sponsoring another session on water at this annual meeting in the form of a Symposium on Problems in the Classification of Natural Water Intended for Industrial Use. It is believed the information that will be made available by the papers and the discussion will permit Subcommittee V on Classification to proceed with the work included in its scope. The following papers[2] are in prospect:

"Reporting the Results of Water Analysis," by Robert C. Adams.

"A Review of Data on the Relationship of Corrosivity of Water to Its Chemical Analysis," by V. V. Kendall.

"Measuring the Scale-Forming and Corrosive Tendencies of Water by Short-Time Tests," by Everett P. Partridge and G. B. Hatch.

"A Method of Measuring Corrosiveness," by J. H. Walker.

The Society has been represented by Max Hecht and E. P. Partridge at two meetings of the Feedwater Subcommittee of the A.S.M.E. Boiler Code Committee. The subcommittee has been considering revisions of Paragraph CA-5 of the Boiler Construction Code.[3] A report by C. W. Rice, chairman of the subcommittee, and the proposed revisions of the paragraph in question have been published in *Mechanical Engineering*[4] under the title "Addenda to Par. CA-5 of Boiler Construction Code."

The subject of hydrogen ion determinations (pH) has been referred by the Executive Committee of the Society to Committee E-1 on Methods of Testing with the recommendation that Committee E-1 undertake the organization of a technical committee to study and develop standard methods of determining hydrogen ion concentration in various materials and to act in an advisory capacity to the several standing committees of the Society which might require information on the subject.

I. New Tentative Standard

The committee recommends that the proposed Tentative Method of Reporting Results of Analysis of Industrial Waters[5] be accepted for publication as tentative. This method has been developed by Subcommittee V on Classification and has been under consideration since 1937.

II. Revisions of Tentative Standards

Constructive comments have been received for the improvement of several of the D-19 methods for analysis of industrial waters, since their publication as tentative in 1938. The revisions, affecting both form and substance, are intended to make the methods generally acceptable. The committee accordingly recommends that the following tentative methods be revised, as given in detail in the Appendix to this report, and continued as tentative:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, pp. 1191, 1204, 1221, 1233, 1242 (1939).

[2] See pp. 1307, 1317, 1329, 1342.

[3] A.S.M.E. Boiler Construction Code, Combined Edition, Sections I to VIII, p. 442 (1935).

[4] *Mechanical Engineering*, March, 1940, pp. 249–252.

[5] This method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 541.

Tentative Methods for:

Sampling Plant or Confined Waters for Industrial Uses (D 510 – 38 T),

Determination of the Calcium Ion and Magnesium Ion in Industrial Waters (D 511 – 38 T),

Determination of the Chloride Ion in Industrial Waters (D 512 – 38 T),

Determination of the Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Industrial Waters (D 515 – 38 T), and

Determination of the Sulfate Ion in Industrial Waters (D 516 – 38 T), completely revised as appended hereto.[6]

III. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The committee recommends that the methods listed below be continued as tentative without revision. No comments have been received concerning them since their publication as tentative in 1938. However, the committee intends to amplify the methods by the addition of field or rapid control procedures, following which it is expected that they will be recommended for adoption as standard:

Tentative Methods for:

Determination of Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Waters (D 513 – 38 T), and

Determination of the Hydroxide Ion in Industrial Waters (D 514 – 38 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 62 members; 49 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II, Editorial (E. P. Partridge, chairman).—No meeting of this subcommittee was held during the year. However, the function of the subcommittee is to assist in preparing committee material for publication with respect to form rather than to substance. It thus operates parallel to and in cooperation with those subcommittees engaged in the formulation of standards. The items comprising the activities of this subcommittee during the past year are briefly discussed in the following several paragraphs.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Method of Reporting Results of Analysis of Industrial Waters	43	3	3
II. REVISIONS OF TENTATIVE STANDARDS			
Sampling Plant or Confined Waters for Industrial Uses (D 510 – 38 T)	47	1	1
Determination of the Calcium Ion and Magnesium Ion in Industrial Waters (D 511 – 38 T)	47	1	1
Determination of the Chloride Ion in Industrial Waters (D 512 – 38 T)	47	0	2
Determination of the Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Industrial Waters (D 515 – 38 T)	47	0	2
Determination of the Sulfate Ion in Industrial Waters (D 516 – 38 T)	47	0	2

A draft of the Benzidine Method for the Determination of Sulfate Ion, originally made available to Committee D-19 through the courtesy of the Joint Research Committee on Boiler Feedwater Studies, was edited with respect to form, and referred to Subcommittee IV, for criticism as to content.

Proposed revisions prepared by Subcommittee III for the tentative methods of sampling industrial waters and by

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[6] This revised method was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 539.

Subcommittee IV for the four tentative methods of analysis of industrial waters were approved as to form.

The proposed Tentative Method of Reporting Results of Analysis of Industrial Waters,[5] developed by Subcommittee V, was reviewed and approved with respect to form.

Subcommittee III on Methods of Sampling (M. A. Krimmel, chairman).—One meeting of the subcommittee was held during the year. Considerable attention has been given to revisions in the methods of sampling (D 510 - 38 T), and as a result, several important changes are being proposed as given in the Appendix.

The study of the present methods has revealed the necessity of cooperating with Subcommittee IV on certain items, particularly with reference to the preparation of samples. Methods of treatment for samples will be developed jointly by Subcommittees III and IV, attention to be given to samples which it is necessary to treat when they are taken or shortly thereafter so that the methods of analysis will not be adversely affected.

The importance of having suitable containers for samples and of resistant materials for coils for cooling hot samples and condensing steam in order to eliminate contamination of the samples has come to the fore. A special subcommittee is investigating materials for containers and cooling coils with the object of eventually being able to specify the most suitable materials for various conditions.

Sampling methods will be developed according to the following outline, based on discussions at meetings and comments made in correspondence:

Part IV–A. Water at atmospheric pressure:

1. Wells and large bodies of water.
2. Pipe lines and conduits.
3. Tanks and vats.
4. Spray ponds and towers.
5. Gravity filters.
6. Water softeners.

Part IV–B. Water at superatmospheric pressure:

1. Pipes and pumps.
2. Fired vessels.
3. Unfired vessels.

Part IV–C. Water at subatmospheric pressures:

1. Pipes and pumps.
2. Steam condensers and heat exchangers.
3. Vacuum deaerators.
4. Vacuum evaporators.
5. Vacuum stills.

Part V. Water in vapor state (steam).

Part VI. Water in solid state (ice).

Part VII. Waste water.

Each of the sections listed above will be developed by a special subgroup of Subcommittee III. Literature in the fields covered by the outline is being reviewed by some of the members and their observations will be reported and used in the development of the methods.

Subcommittee IV on Methods of Analysis (H. C. Miller, chairman).—This subcommittee held one meeting during the year. It is recommending that four tentative methods of analysis of industrial waters be revised and continued as tentative, as mentioned earlier in this report.

During the current year work on additional methods of analysis has been carried forward and a preliminary draft of a method for the determination of silica has been distributed to subcommittee members for criticism. Methods for the determination of iron, aluminum, manganese, and hardness are in preparation and will soon be available for consideration.

Subcommittee IV has had the active cooperation of Subcommittee III in preparing preliminary drafts of the referee and field methods for the determination of dissolved oxygen, particularly those sections dealing with sampling, and these will soon be available. Material is now being collected for the preparation of routine methods for sulfates, calcium, magnesium, phosphates, and sulfites.

A canvass of the members for data on additional methods not so far considered by the subcommittee indicates that material will shortly be available for the preparation of methods for the determination of dissolved solids by evaporation and conductivity, the routine determination of organic matter, and both referee and rapid procedures for suspended matter, sodium, and ammonium.

Subcommittee V on Classification (R. C. Adams, chairman).—This subcommittee held one meeting during the year. After two complete revisions agreement has been reached by the Subcommittee on a proposed Tentative Method of Reporting Results of Water Analysis,[5] which is being recommended for publication as tentative. This report is the result of the Round-Table Discussion on Interpretations of Water Analyses from Analytical Data held in New York City in 1937, at which time action was taken to organize a subcommittee on classification, and to initiate work on a standard method of reporting a water analysis.

The subcommittee chairman has cooperated with the members of the Advisory Committee of Committee D-19 in arranging the symposium which is to be held at the 1940 annual meeting of the Society, mentioned earlier in this report. The paper to be presented by the subcommittee chairman at the symposium is intended to focus the attention of engineers as well as chemists who deal in water problems on the desirability and necessity of including all potential data in the report of the analysis, and to provide a convenient and simple procedure for reporting the results.

The election of officers resulted in the continuance in office of the present incumbents for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 62 members; 49 members returned their ballots, of whom 44 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. E. HALL,
Secretary.

APPENDIX

PROPOSED REVISIONS IN STANDARDS FOR INDUSTRIAL WATERS

In this Appendix are given proposed revisions in certain tentative methods of analysis of industrial waters which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the method appears in its present form.

Tentative Methods of Sampling Plant or Confined Waters for Industrial Uses (D 510 - 38 T):[1]

Table I.—Under Physical Tests, omit the word "Total" from the requirement for "Total dissolved solids," and change the requirement for volume in milliliters of the sample for color and odor tests from "50 to 1000" to read "100 to 500." Under Chemical Tests, change the subheading "Metallic Ions" to read "Metallic Ions (Cations);" also, change the subheading "Nonmetallic Ions" to read "Nonmetallic Ions (Anions)." Following the table, add a footnote "*a*" referring to the volume of sample, to read as follows:

Volume specified in this table should be considered as a guide for the approximate (usually maximum) quantity of sample necessary for the particular analysis. The exact quantity used should be consistent with the volume prescribed in the standard method of analysis, whenever the volume is specified.

Section 8 (b).—Insert the following paragraph at the beginning of the note:

Since new corks are frequently bleached with oxalic acid, it is advisable to rinse them thoroughly with the water sampled.

Insert the following paragraph just preceding the present last paragraph of the note:

In case glass introduces contaminants into solution, a suitable container may be used, but both container and closure shall be of the same material.

Section 13.—Add the following as Note 1, renumbering the present note as Note 2:

Note 1.—Cooling to as near 70 F. (21 C.) as possible is particularly desirable when the oxygen content of the sample is to be determined, since the sensitivity of the starch indicator solution decreases with increase in temperature.

Add the following paragraphs to the present note:

Since in this procedure the coil is under the pressure existing in the apparatus, it is necessary that the coil be built to stand the required pressure with a reasonable margin of safety.

Samples should be cooled by water under less pressure than the water being sampled, or the cooling coil should be designed with a continuous through coil for the sampled water. The cooling water jacket should be sealed against the sample water tube by packing glands rather than direct connections. The bore of the tube should be as small as possible, to allow a minimum hold-up of sample and a maximum of surface for cooling.

Section 15 (b).—Change to read as follows by omission of the words in brackets:

(*b*) Stoppers [,whether corks or glass,] shall be wired, tied, or taped down to prevent leakage in transit.

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[1] 1939 Book of A.S.T.M. Standards, Part III, p. 1096.

Tentative Method for Determination of the Calcium Ion and Magnesium Ion in Industrial Waters (D 511 - 38 T):[2]

Section 1.—Change the last sentence to read as follows by the addition of the italicized word:

> Directions for the preliminary removal of silica, *phosphates*, iron, and aluminum are given, together with special directions for the removal of manganese, when necessary.

Section 3 (e).—Change to read as follows by the addition of the italicized material and the omission of that in brackets:

> (*e*) Diluted Ammonium Hydroxide [(1:10)] *(1:20)*.—Prepare a diluted solution of NH_4OH by adding one volume of reagent grade NH_4OH (sp. gr. 0.90) to [ten] *twenty* volumes of distilled water.

Delete the description of the diluted ammonium hydroxide solution (1:50) in Section 3 (*f*), relettering the subsequent paragraphs accordingly.

To conform with the revisions in Section 3, recommended above, change Sections 6 (*d*), 7 (*c*), 10 (*c*), and 11 (*b*) to provide for the use of NH_4OH (1:20) only.

Section 4.—Change from its present form to read as follows:

> 4. The volume of water sample used for the determination of calcium and magnesium shall preferably contain 10 to 100 mg. of calcium.

Section 5 (a).—Change from its present form: namely,

> 5. (*a*) *Removal of Silica.*—The measured volume of the water shall be acidified by adding 1 to 2 ml. of HCl (sp. gr. 1.19), and shall then be evaporated to dryness.

to read as follows:

> 5. (*a*) The sample shall be acidified with HCl (sp. gr. 1.19), adding 2 ml. in excess, and then shall be evaporated to dryness.

Retain the note following this paragraph in its present form.

Section 6.—Change the title of Paragraph (*a*) to read as follows by the addition of the italicized word: "Removal of *Phosphates*, Iron, Aluminum, and Manganese." Add a new Paragraph (*c*) to read as follows, relettering the subsequent paragraphs accordingly:

> (*c*) *Removal of Phosphates.*—If phosphates are shown by preliminary test to be present, sufficient iron in the form of a soluble ferric salt of reagent grade (preferably chloride) shall be added to the combined filtrate and washings from Section 5 (*c*) to give a sufficient excess to produce a red-brown precipitate, after which the procedure described in Paragraph (*d*) or (*e*) shall be followed.

Reletter Paragraph (*c*) as (*d*) and change the first sentence to read as follows by addition of the italicized material:

> The combined filtrate and washings procured under Section 5 (c) *or Paragraph (c) of this section* shall be heated to boiling and a few drops of HNO_3 (sp. gr. 1.42) shall be added.

Section 10.—Insert the following note after Paragraph (*a*):

> NOTE.—Diammonium hydrogen orthophosphate may be used as a precipitant in place of sodium ammonium hydrogen orthophosphate, if preferred.

Section 11 (c).—Change from its present form: namely,

> (*c*) *Ignition of Precipitate.*—The filter paper and its contents shall be dried and then ignited to constant weight.

to read as follows:

> (*c*) The filter paper and contents shall be placed in a weighed crucible and thoroughly dried, after which the paper shall be slowly charred and consumed without inflaming. The residue shall then be ignited to constant weight.
>
> NOTE.—Unless ignition of paper and precipitate is done slowly and at a low temperature until carbon is consumed, the carbon becomes partially "fireproofed" and cannot be burned out.

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[2] 1939 Book of A.S.T.M. Standards, Part III, p. 1101.

Tentative Method for Determination of the Chloride Ion in Industrial Waters (D 512 - 38 T):[3]

Section 3.—Delete Paragraph (*d*), relettering Paragraph (*e*) as (*d*).

Section 5.—Change from its present form to read as follows:

> 5. *Removal of Turbidity or Color.*—If the turbidity or color, or both, of the sample is so great as to obscure or render uncertain the potassium chromate indicator end point, acidify the 50-ml. sample by adding 5 ml. of reagent grade 3 *N* H_2SO_4. Heat to boiling and filter, neutralize with reagent grade 5 *N* NaOH to phenolphthalein end point, and then just destroy the pink color with reagent grade 0.1 *N* H_2SO_4.

Section 7.—Change Paragraph (*b*) from its present form: namely,

> (*b*) Each of the two portions shall be separately adjusted to a pH of 9.0 ± 0.2, using solutions of reagent grade H_2SO_4 or Na_2CO_3 of suitable concentration as required. The pH may be measured either colorimetrically with a comparator or electrometrically with an apparatus which does not include a salt bridge.

to read as follows:

> (*b*) Each of the two portions shall be separately adjusted to a pH of 8.3 ± 0.5 using solutions of reagent grade H_2SO_4 or NaOH of suitable concentration, as required.
>
> NOTE.—Sufficiently accurate adjustment of the required pH may be obtained by making the solution just alkaline to phenolphthalein by adding reagent grade NaOH and then just destroying the pink color with reagent grade H_2SO_4.

Tentative Method for Determination of the Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Industrial Waters (D 515 - 38 T):[4]

Section 4.—Add the following as new Paragraph (*a*), relettering the subsequent paragraphs accordingly:

> (*a*) To a quantity of the sample greater than required in the determination, a quantity of phosphate-free reagent grade KNO_3 sufficient to give approximately a 4 per cent solution shall be added. The solution shall be heated to boiling, and shall then be removed from the source of heat and allowed to stand for a few minutes in order to settle the coagulated material. The solution shall then be filtered.

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[3] 1939 Book of A.S.T.M. Standards, Part III, p. 1105.

[4] 1939 Book of A.S.T.M. Standards, Part III, p. 1113.

REPORT OF COMMITTEE D-20

ON

PLASTICS

Since the June, 1939, meeting in Atlantic City, N. J., Committee D-20 on Plastics and its subcommittees have held two meetings: one in New York, N. Y., in October, 1939, and one in Washington, D. C., in February, 1940. The committee expects to hold a further meeting in Atlantic City during this annual meeting.

L. M. Currie has resigned as chairman of Subcommittee III and W. A. Zinzow has been appointed in his place; H. W. Paine has resigned as chairman of Subcommittee IV and W. F. Bartoe has succeeded him.

The following officers were unanimously elected at the February meeting to serve from June, 1940, to June, 1942:

Chairman, W. E. Emley.

First Vice-Chairman, Bjorn Andersen.

Second Vice-Chairman, L. M. Currie.

Secretary, W. A. Evans.

Committee D-20 now has a membership of 62 voting members, representing an increase of 4 voting members.

NEW TENTATIVE STANDARDS

The committee recommends that the following three methods of test be accepted for publication as tentative:

Tentative Methods of:[1]

Test for Flammability of Plastics,

Measuring Flow Temperatures of Thermoplastic Molding Materials, and

Test for Water Absorption of Plastics.

These recommendations have been submitted to letter ballot of the committee which consisted of 58 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
NEW TENTATIVE STANDARDS			
Test for Flammability of Plastics	32	0	18
Measuring Flow Temperatures of Thermoplastic Molding Materials	29	0	10
Test for Water Absorption of Plastics	36	0	13

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (M. H. Bigelow, chairman) is studying the technique and the methods of compression, tension, and impact testing of plastics. Particular study is being made of the tension specimen for thermoplastics.

Subcommittee II on Hardness Properties (J. C. Pitzer, chairman) has under way a series of tests using a proposed deformation under load method. Considerable progress has been made in the study of mar, scratch, wear, and abrasion resistance.

Subcommittee III on Thermal Properties (W. A. Zinzow, chairman) is giving its attention to studies of heat and fire resistance, and flow and molding characteristics. A method designed to determine the facility with which plastic materials in thin sheet or film form will burn has been developed because of the many disagreements as to the rapidity with which such burning takes place. If the tendency of a plastic to burn is measured in exactly the same way by all parties at interest, the present disagreements on this point will disappear. The new method, presented for publication as tentative as mentioned earlier in this report, is limited to thin films not more than 0.05 in. in thickness.

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[1] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 284, 286, and 292, respectively.

The method for measuring flow temperatures of thermoplastic molding materials was developed in order that the seller and the purchaser of molding powders would have some means of determining this important property. The method will also be found of use to the purchaser of such molding powders to enable him properly to control the molding process. The method, presented to the Society for publication as tentative as mentioned earlier in this report, has been developed from studies reported in a paper on "Measurement of the Flow Temperature of Thermoplastic Molding Materials," by L. W. A. Meyer.[2]

Subcommittee IV on Optical Properties (W. F. Bartoe, chairman) is studying the following optical characteristics: haze, reflection factors, polarization, surface irregularities, and surface brightness. A test method for measuring surface irregularities of transparent organic plastics is in preparation.

Subcommittee V on Permanence Properties (G. M. Kline, chairman) is carrying on studies of the resistance of plastics to heat, light, and moisture, and the effect on other characteristics. Cooperative work with Committee D-9 on Electrical Insulating Materials has resulted in the development of a generally applicable Method of Test for Water Absorption of Plastics, which is being presented for publication as tentative, as mentioned earlier in this report. Studies forming a basis for the development of this method are described in a paper by G. M. Kline, A. R. Martin, and W. A. Grouse, "Sorption of Water by Plastics."[3]

This report has been submitted to letter ballot of the committee which consists of 62 voting members; 50 members returned their ballots, of whom 48 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. E. EMLEY,
Chairman.

W. A. EVANS,
Secretary.

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[2] Published in ASTM BULLETIN, No. 105, August, 1940, p. 23.

[3] See p. 1273.

EDITORIAL NOTE

Subsequent to the annual meeting Committee D-20 presented to the Society through Committee E-10 on Standards a revision of the Tentative Methods of Test for Index of Refraction of Transparent Organic Plastics (D 542 – 39 T). The tentative methods in their revised form appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 289.

REPORT OF COMMITTEE E-1

ON

METHODS OF TESTING

Increasing interest in the work of this administrative Committee on Methods of Testing on the part of the standing committees of the Society has been evidenced by the requests received during the past year which have resulted in several new projects being undertaken by the committee.

These subjects as well as the activities of the committee during the year are briefly summarized in this annual report.

A meeting of the Advisory Committee was held in Philadelphia, Pa., on May 28, 1940, and meetings of several of the technical committees and sections were held in Detroit, Mich., in March, 1940, in connection with the spring group meetings of the Society. A number of the technical committees and sections are also holding meetings during this annual meeting.

The Executive Committee of the Society appointed three representatives-at-large to serve on the Advisory Committee, namely, L. H. Fry, Edgewater Steel Co., D. E. Parsons, National Bureau of Standards, and T. Smith Taylor, Newark College of Engineering, each for a three-year term, expiring in 1942.

Subsequent to the 1939 annual meeting, Committee E-1 presented to the Society through Committee E-10 on Standards a tentative revision of the Standard Methods of Rockwell Hardness Testing of Metallic Materials (E 18 – 36), in the form of a new Tentative Method of Test for Rockwell Superficial Hardness of Metallic Materials (E 18 – 39 T). This tentative revision was accepted[1] by Committee E-10 on October 23, 1939, and appears in the 1939 Book of A.S.T.M. Standards, Part I.

Accelerated Weathering Tests.—A conference of representatives of the standing committees interested in the subject of accelerated weathering tests was held under the auspices of Committee E-1 at the 1939 annual meeting of the Society with E. F. Hickson serving as chairman. Representatives of the following committees presented their views on the subject: D-1 on Paint, D-4 on Road and Paving Materials, D-8 on Bituminous Waterproofing and Roofing Materials, D-9 on Electrical Insulating Materials, D-13 on Textile Materials, and D-20 on Plastics. This was followed by an interesting general discussion of accelerated weathering tests by members of a number of Society committees and others present, the attendance being approximately 100. The conference resulted in the recommendation that a correlating committee on this subject be formed to provide a means of contact and coordination of the work of committees of the Society dealing with studies of accelerated weathering tests. This recommendation was considered by the E-1 Advisory Committee at its May meeting and received the approval of that committee.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee E-1 reported results of the letter ballot vote as follows: Of a total membership of 52 45 members returned their ballots, of whom 31 voted affirmatively, 0 negatively, and 14 marked their ballots "not voting."

In considering the organization of this work it was recognized that the necessary machinery for coordinating the efforts of the standing committees in the accelerated weathering field might well be provided for within the existing structure of Technical Committee X on Conditioning. The membership of this technical committee includes representatives of the standing committees interested in this class of endeavor, and such additions to the personnel as may be necessary can be readily made. The Advisory Committee gave favorable consideration to this means of initiating the work and has recommended that Technical Committee X form a Section on Accelerated Weathering Tests for the coordination of activities in this field.

Hydrogen Ion Determinations.—During the year there was referred to Committee E-1 by the Executive Committee of the Society a recommendation that Committee E-1 undertake the organization of a technical committee on the subject of hydrogen ion determinations, having for its purpose the study of standard methods of determining hydrogen ion concentration in various materials and acting in an advisory capacity to the several standing committees of the Society that might require information on the subject.

The recommendation of the Executive Committee was considered favorably by the Advisory Committee and the organization of a new Technical Committee on Hydrogen Ion Determinations was authorized.

I. New Tentative Standards

The committee recommends that the following be accepted for publication as tentative:

Tentative Methods of Tension Testing of Metallic Materials,[2] to replace immediately the present Standard Methods of Tension Testing of Metallic Materials (E 8 – 36), and

Tentative Recommended Practices for Designation of Numerical Requirements in Standards.[3]

II. Revisions of Tentative Standards

The committee recommends that the following tentative standards be revised and continued as tentative:

Tentative Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 37 T), revised as appended hereto,[4] and

Tentative Specifications for A.S.T.M. Thermometers (E 1 – 39 T), by the addition of requirements for the turpentine distillation thermometer,[5] which comprise a revision of the specifications for thermometer E 1 (27 C – 39) in Standard E 1 – 39.[6]

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 52 members; 48 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Tension Testing of Metallic Materials (E 8 – 40 T), to replace immediately Standard Methods E 8 – 36	34	0	14
Recommended Practices for Designation of Numerical Requirements in Standards	40	0	8
II. REVISIONS OF TENTATIVE STANDARDS			
Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 37 T)	30	2	16
Spec. for A.S.T.M. Thermometers (E 1 – 39 T), revision of turpentine distillation thermometer E 1 (27 C – 39)	30	0	18

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[2] These methods were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 453.

[3] These recommended practices were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 464; Part II, p. 325; Part III, p. 549.

[4] These definitions were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 472; Part II, p. 333; Part III, p. 557.

[5] The revision was accepted by the Society and has been incorporated in the specifications which appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 321; Part III, p. 545.

[6] 1939 Book of A.S.T.M. Standards, Parts II and III.

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee I on Mechanical Testing (H. F. Moore, chairman):

The work of this committee is best described by brief summaries of the work of its sections.

Section on Tension Testing (R. L. Templin, chairman).—During the year the section has continued work on the revision of the Standard Methods of Tension Testing of Metallic Materials (E 8 – 36). An extensive revision has been prepared, and has received approval by letter ballot of this section as well as of Technical Committee I. It is accordingly recommended that the revised methods be accepted for publication as tentative[2] to replace immediately the present Standard Methods E 8 – 36.

The section held a meeting in Detroit, Mich., on March 6, 1940, at which the effect of speed of testing in tension tests, the "proof stress" required in certain U. S. Government specifications, and other matters of import to the section were discussed.

This section cooperated through its chairman in arranging for the Symposium on Significance of the Tension Test of Metals in Relation to Design[7] to be held at this annual meeting.

Section on Compression Testing (M. F. Sayre, chairman).—This section held a meeting in Atlantic City, N. J., June 27, 1939, at which the question was reopened of revising the Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T). Consideration was given to compression tests under the following four divisions: (*1*) tests of brittle materials for ultimate strength, (*2*) tests of ductile materials for yield strength, (*3*) tests of thin sheet, ductile and brittle, by the pack method, and (*4*) tests of bearing metals for behavior during plastic flow. This study has been continued during the year and a revision of Tentative Methods E 9 – 33 T is in preparation.

Section on Bend Tests for Ductility (A. B. Kinzel, chairman).—In view of the fact that the immediate work of this section was completed last year by the adoption as standard of the Method of Bend Testing for Ductility of Metals (E 16 – 39), the section has recommended that it be discharged. This recommendation was approved by the Advisory Committee at its May meeting.

Section on Indentation Hardness Testing (J. R. Townsend, chairman).—The Tentative Method of Test for Rockwell Superficial Hardness of Metallic Materials (E 18 – 39 T), which was accepted by the Society through Committee E-10 on Standards, as mentioned earlier in this report, was prepared under the auspices of this section.

A special committee of this section has been appointed to work with the Subcommittee on Thermostat Metals of Committee B-4 on Electrical Heating, Electrical-Resistance and Electric-Furnace Alloys for the purpose of developing hardness requirements for thermostat metals. The special committee will consist of the following: C. H. Davis of the American Brass Co., V. L. Lysaght of the Wilson Mechanical Instrument Co., and A. Sonntag of Riehle Brothers Testing Machine Division of American Machine and Metals, Inc.

Section on Testing Thin Sheet Metals (C. H. Marshall, chairman).—This section has recently taken up the study of test specimens for strip steel. The question has been raised whether pieces of narrow strip cut from a coil can be used as test specimens to give reliable tension test results without reducing the width over the middle portion of the length.

Section on Impact Testing (W. W. Werring, chairman).—At the meeting of this section held in Atlantic City, N. J., on June 27, 1939, a proposed revision of the Tentative Methods of Impact Testing of Metallic Materials (E 23 – 34 T) was discussed. This revision is still being studied and will be given detailed consideration by the section at its meeting during this annual meeting.

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[7] See pp. 501 to 609, inclusive.

Section on Effect of Speed of Testing (P. G. McVetty, chairman).—A meeting of this section was held in Detroit, Mich., on March 6, 1940, at which a preliminary report was presented on the investigation of the effect of speed of testing on the properties of metals, being conducted at the University of Illinois by Paul G. Jones and H. F. Moore. At this meeting also there was presented by Lawford H. Fry[8] a report of studies made under the auspices of Subcommittee XIII on Methods of Physical Tests of Committee A-1 on Steel. The two reports pointed toward similar conclusions, and gave ground for hope that a simpler method of study of speed effect might be developed on the basis of the two reports, and that feasible methods of specifying rates of strain in mechanical tests might be developed. The report on the studies at the University of Illinois, referred to above, is being presented at this annual meeting as a technical paper by Paul G. Jones and H. F. Moore, "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals."[9]

This section is considering the possibility of investigating the effect of speed of testing of some nonmetallic materials.

Section on Elastic Strength of Materials (F. B. Seely, chairman).—A meeting of this section is to be held during this annual meeting of the Society at which specifications for a "proof stress" which may serve as a measure of elastic strength will be considered. This matter has been brought to the attention of the section through the use of such a "proof stress" in certain U. S. Government specifications.

Section on Calibration of Testing Machines and Apparatus (H. F. Moore, chairman).—This section will hold a meeting during this annual meeting of the Society at which the calibration of strain-measuring apparatus will be considered.

Technical Committee II on Consistency, Plasticity, and Related Properties (E. C. Bingham, chairman):

This technical committee held a meeting in Atlantic City, N. J., on June 28, 1939, at which the comments that had been received on the Tentative Definitions of Terms Relating to Rheological Properties of Matter (E 24 – 37 T) were discussed at some length. The definitions were extensively revised at this meeting and subsequently approved by letter ballot vote of the technical committee. The revised definitions are being presented for publication as tentative,[4] as referred to earlier in this report.

A recommendation has been received from Committee D-17 on Naval Stores that the Tentative Method of Test for Softening Point (Ball and Tapered Ring Apparatus) (E 28 – 39 T) be further revised to permit the optional use of a shouldered ring for testing certain high melting resins, modified rosins, and other products which so contract on cooling that they tend to slip part way out of the tapered ring. This recommendation together with a drawing of the proposed shouldered ring and a report containing comparative results for both types of rings in tests on several types of resins has been transmitted to the members of the Special Committee on Softening Point Test, under the chairmanship of T. H. Rogers, and is now receiving consideration by that group.

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[8] Lawford H. Fry, "Speed in Tension Testing and Its Effect on Yield Point Values," see p. 625.

[9] See p. 610.

Some consideration is being given by this technical committee to the establishment of a set of symbols for use in connection with rheologic terms.

A proposal has recently been referred to the committee by the chairman for a cooperative investigation into the fundamental properties of flow. This would include a study of the methods of testing materials involving the flow of those materials, for the purpose of ascertaining whether the results obtained by the methods now in use can be expressed in fundamental units of flow and eventually correlated with each other and with any other methods that may be devised, to obtain values that are more simple, rapid, precise, or significant.

Technical Committee III on Particle Size and Shape (L. T. Work, chairman):

This technical committee has been reorganized into the following sections:

Advisory, L. T. Work, chairman,
Sieves, A. A. Klein, chairman,
Pigment-Type Materials, C. E. Barnett, chairman, and
Ground Products, L. T. Work, chairman.

F. H. Jackson has been appointed as vice-chairman and L. V. Judson as secretary of the technical committee.

The personnel of the sections has been rearranged to conform to the several fields of interest and the sections are closely watching their respective fields. Work is actively in progress in the Section on Pigment-Type Materials. It was hoped that a major revision of the Tentative Method of Test for the Particle Size Distribution of Subsieve Size Particulate Substances (E20–33T), devoted only to pigment-type materials, would be available for action at this time, but the section feels that although work on the method is well advanced, further modification is needed to make it acceptable to all concerned. It is expected that the revised method will be presented for publication next year.

The Section on Pigment-Type Materials is sponsoring a Symposium on Particle Size Determination to be held at the 1941 Spring Meeting of the Society in Washington, D. C., and several excellent papers have already been obtained. It is hoped that this symposium will serve as a guide in the formulation of new standards in the subsieve range.

The Sections on Sieves and on Ground Products are merely watching developments and contemplate no immediate efforts in the development of new standards.

Technical Committee IX on Interpretation and Presentation of Data (H. F. Dodge, chairman):

During the past year this technical committee has served in an advisory capacity to several standing committees of the Society on problems relating to the use of statistical methods in the analysis and presentation of data.

The committee held a meeting in Detroit, Mich., on March 6, 1940, at which plans were discussed for revising the A.S.T.M. Manual on Presentation of Data. The revision will include the addition of a supplement on the subject of sampling errors of averages. The supplement is to treat not merely the statistical theory that applies but is to consider practical engineering procedures that may be followed where the assumptions and conditions of theory cannot be assumed to hold.

Technical Committee X on Conditioning (Robert Burns, chairman):

The members of Technical Committee X have approved, by letter ballot vote, the scope of the committee,[10] and the following definition of conditioning which was proposed at a previous meeting:

> Conditioning is the process of subjecting a material to a stipulated influence or combination of influences for a stipulated period of time. The influences may be relative humidity, temperature, light, pressure, air composition, or other factors, used jointly or severally.
>
> NOTE 1.—Conditioning may be used for (*a*) predicting service behavior, (*b*) obtaining reproducible results, (*c*) testing for acceptance, (*d*) research, (*e*) process control; for any single objective or combination of objectives.
>
> NOTE 2.—It is generally true that only a portion of the influences enumerated in the above definition are applicable to any given material. Hence in any discussion of conditioning the particular influences that are significant must be stated.

As part of its function to encourage, wherever possible, the utilization of existing conditioning methods, Technical Committee X has given wide circulation to a brief outline of the more extensively used combinations of atmospheric influences now employed in the testing of materials, including those which have been made part of existing standards and those which are not standard but have found extensive application. A number of replies have been received from various industrial organizations informing the committee of their adoption of the recommendations.

The committee is reviewing the definitions included in the Standard Method of Determining Relative Humidity (D 337 – 34), under the jurisdiction of Committee D-13 on Textile Materials, and also the definition of average room conditions, prepared by Committee D-9 on Electrical Insulating Materials, with a view to determining their suitability for general adoption.

Several new conditioning procedures now under active consideration of the standing committees are being prepared with the assistance of members of this technical committee. Prominent among these projects is the work of Committee D-9 on the development of a standard conditioning chamber.

Technical Committee XI on Designation and Interpretation of Numerical Requirements (J. A. Gann, chairman):

This technical committee held a meeting in Detroit, Mich., on March 6, 1940, at which further consideration was given to comments received on the Proposed Recommended Practices for Designation of Numerical Requirements in Standards.[3] The question of the method of rounding off decimal values, which has been the subject of some considerable discussion in the committee, has been solved by incorporating a rounding off procedure that is in agreement with that covered by the Proposed American Standard Rules for Rounding Off Numerical Values. The proposed recommended practices have been approved by letter ballot vote of the technical committee and are being presented for publication as tentative, as mentioned earlier in this report.

Technical Committee XII on Laboratory Apparatus (W. H. Fulweiler, chairman):

The activities of this technical committee have been handled by its several sections. Brief summaries of the work of the active sections are presented below.

Section A on Thermometers (R. M. Wilhelm, chairman).—Revised specifications for the turpentine distillation thermometer have been prepared,[5] as mentioned earlier in this report, and are recommended for publication as a tentative revision of the present Standard Specifications for A.S.T.M. Thermometers (E 1 - 39).

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[10] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 524 (1939).

In cooperation with Committee D-2 on Petroleum Products and Lubricants the section has also prepared specifications for three aniline point thermometers, to be published as information in the Proposed Method of Test for Aniline Point of Petroleum Products.[11]

Consideration is being given by the section to specifications for the titer test thermometer for use in the testing of soap.[12]

A review of the Standard Specifications for A.S.T.M. Thermometers (E 1 - 39) has resulted in the approval of several editorial changes which will be incorporated in the specifications when they are next printed.

Section C on Volumetric Glassware (J. J. Moran, chairman).—This section has cooperated with several of the standing committees in reviewing drafts of new and revised methods containing requirements for calibrated glass apparatus, and as a result has made recommendations for improvements in certain features of the apparatus.

A paper on "Methods of Testing Volumetric Glassware" by the chairman of this section was contemplated for presentation to the Society at this annual meeting. It has not, however, been found possible to complete the paper this year.

Section D on Laboratory Glassware (F. F. Shetterly, chairman).—This section also has reviewed and presented suggestions for improvements in requirements for certain pieces of laboratory glassware appearing in new and revised methods being submitted to the Society this year by other standing committees.

Section E on Ceramic Apparatus (H. W. Ryland, chairman).—This section was organized during the year and consists of the following members:

H. W. Ryland, chairman,
C. G. Amend,
B. L. Clarke,
J. R. Dwyer,
H. C. Fisher,
J. A. Hartley, and
E. O. Rhodes.

Section F on Metalware Apparatus (J. E. Patterson, chairman).—An organization meeting of this section was held in Washington, D. C., on March 13, 1940. The membership of the section is as follows:

J. E. Patterson, chairman,
A. W. Fisher,
C. A. Hogentogler,
M. F. Kleeberg,
P. E. Klopsteg,
W. W. Pitann,
C. S. Reeve,
W. H. Reynolds,
W. A. Selvig,
R. M. Wilhelm, and
L. G. Wilson.

The programs of work to be undertaken by the two new sections will be considered during the year.

W. H. Fulweiler and H. F. Moore have been re-elected chairman and vice-chairman, respectively, of Committee E-1 for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 52 members; 48 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. FULWEILER,
Chairman.

P. J. SMITH,
Ex-officio Secretary.

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[11] See p. 313.

[12] Proposed requirements for this thermometer appear in the Report of Committee D-12 on Soaps and Other Detergents, see p. 399.

REPORT OF COMMITTEE E-2 ON SPECTROGRAPHIC ANALYSIS

Committee E-2 on Spectrographic Analysis held a meeting in Atlantic City, N. J., on June 29, 1939. A further meeting will be held during this annual meeting.

The committee sponsored a round-table discussion on Fundamental Methods and Technique of Spectrochemical Analysis at the June, 1939, meeting of the Society. Two well-attended sessions brought attention to bear on several of the fundamentals of the spectrographic method and served as a basis for planning a Symposium on Spectrographic Analysis to consider accuracy requirements, reproducibility possibilities and interpretation of results, to be held at the 1940 annual meeting of the Society. It is planned to publish a coordinated account of the 1940 symposium in the ASTM BULLETIN.

Seven new members were elected to the committee during the year, resulting in a total membership of 60.

Subcommittee II on Fundamental Methods and Technique (O. S. Duffendack, chairman) organized the round-table discussion at the 1939 meeting and held a meeting of the subcommittee following the symposium. During the meeting a survey was made of the situation with regard to the methods that have been developed for spectrochemical analysis and the matter was discussed informally. The desirability for a standardization of the terminology in this field was pointed out.

Members of the subcommittee were active during the year in developing and keeping in contact with new methods for analyzing specific materials. As a step toward consideration of fundamentals of spectrochemical analysis, reports of a basic research on excitation of mercury vapor by electron impacts were circulated among the members of the subcommittee. The subcommittee has recommended that further research of this fundamental nature be encouraged in order to develop a more complete understanding of the excitation processes and fundamental principles of analytical procedures.

Subcommittee III on Quantitative Methods and Applications (M. L. Fuller, chairman) was active during the past year through the work of its several groups. Mr. P. A. Leichtle was appointed chairman of Group 1 on Copper, Nickel and Their Alloys to succeed C. H. Davis, resigned. Group 1 made an initial survey of the practice of spectrographic analysis of copper and nickel base materials, by means of a questionnaire and at a meeting of the group in New York City on March 29, 1940. The discussion at this meeting led to the opinion that the group should make preliminary tests on one or two samples of copper and brass for standards before planning any extensive program of group testing. Steps have been taken to carry out the preliminary testing.

Group 2 on Lead, Tin, Antimony, Bismuth and Their Alloys (A. E. Ruehle, chairman) is engaged in the preparation of methods for the spectrographic analysis of lead and tin.

Group 4 on Zinc, Cadmium and Their Alloys (M. L. Fuller, chairman) developed a revision of the Tentative Method of Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities. The subcommittee approved by letter ballot vote a recommendation that the revised methods be presented to Committee E-2 for submission to the Society to replace Methods E 27 – 37 T.[1]

Subcommittee V on Standards and Pure Materials (T. A. Wright, chairman) handled numerous inquiries as to availability and types of standards required for spectrographic purposes. So far as is known, no new material suitable for standards has been made available, although developments in materials of this type are being followed.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 60 members; 49 members returned their ballots, of whom 47 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. V. CHURCHILL,
Chairman.

B. F. SCRIBNER,
Secretary.

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[1] See Editorial Note below.

EDITORIAL NOTE

Subsequent to the annual meeting Committee E-2 presented to the Society through Committee E-10 on Standards a revision of the Tentative Method of Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 37 T). This recommendation was accepted by Committee E-10 on November 5, 1940, and the revised tentative method has been published separately as a 1940 Supplement to the Methods of Chemical Analysis of Metals.

REPORT OF COMMITTEE E-3
ON
CHEMICAL ANALYSIS OF METALS

Committee E-3 on Chemical Analysis of Metals held one meeting during the year on June 28, 1939, in Atlantic City, N. J., during the annual meeting of the Society. Six division and six subcommittee meetings were also held during the year. The committee is pleased to report that considerable progress has been made during the year.

Committee E-3 took an active part in arranging for this annual meeting the Symposia on Tools of Analytical Chemistry, and on Spectrographic Analysis, as well as the presentation of moving and still pictures illustrating applications of spectrochemical and microanalytical procedures in industry. These meetings attracted a number of guests, drawn from the American Chemical Society, and from industrial and educational laboratories.

Seven new members were elected to the committee during the year, and one member resigned, leaving a total membership of 66.

The present officers of the committee were re-elected for the ensuing term of two years.

Division A on Ferrous Metals (Arba Thomas, chairman) has under consideration methods for the determination of lead and small amounts of aluminum in carbon steel which have been prepared and tested and are ready for letter ballot action by the division.

Subcommittee A-2 on Metallic Materials for Electrical Heating (T. R. Cunningham, chairman), of Division A, is engaged in preparing methods for the analysis of electrical heating alloys. Methods for determining columbium and selenium are being studied by special subcommittees of which W. D. Brown and E. R. Vance are chairmen, respectively.

Division B on Non-Ferrous Metals (R. P. Nevers, chairman) has been active through several of its subcommittees:

Subcommittee B-1 on Copper and Nickel and Their Alloys (R. P. Nevers, chairman) is continuing the extensive work on the preparation of methods for the analysis of copper-base alloys.

Subcommittee B-2 on Lead, Tin, Antimony, Bismuth and Their Alloys (J. B. Mosley, chairman) has completed the preparation of methods for analysis of pig lead and these methods are ready for letter ballot action by the division.

Subcommittee B-4 on Zinc and Its Alloys (R. W. Coltman, chairman) has completed methods for the determination of cadmium, lead and iron in slab zinc and submitted them to the division for action by letter ballot.

Division C on Sampling (T. A. Wright, chairman) through its Subcommittee C-3 (F. M. Barry, chairman) is developing a program for sampling mechanically worked metals.

Division D on General Analytical Methods (B. L. Clarke, chairman), reviewed methods submitted by the ferrous and non-ferrous divisions for the analysis of copper-base and zinc-base alloys; aluminum and lead in carbon steel; and electrical heating alloys of the 80 per cent nickel, 20 per cent chromium type.

This report has been submitted to letter ballot of the committee which consists of 66 members; 65 members returned their ballots, of whom 61 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. E. F. LUNDELL,
Chairman.

H. A. BRIGHT,
Secretary.

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY

Committee E-4 on Metallography has held two meetings during the past year: one in Atlantic City, N. J., on June 28, 1939, during the annual meeting of the Society, and the other in Detroit, Mich., on March 5, 1940.

Subsequent to the 1939 annual meeting Committee E-4 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Standards:

Methods of Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (E 2 – 39 T), to replace immediately Standard Rules E 2 – 36, and

Classification of Austenite Grain Size in Steels (E 19 – 39 T), to replace immediately both the Standard and Tentative Grain Size Charts for Classification of Steels (E 19 – 33 and E 19 – 38 T).

These recommendations were accepted[1] by Committee E-10 on August 24, 1939, and the new tentative standards appear in the 1939 Book of A.S.T.M Standards.[2]

The interest and activities in certain phases of metallographic work have led to the organization of an informal Round-Table Discussion on the Preparation of Metallographic Specimens by Electrolytic Polishing and by Lapping which is to be held during this annual meeting.

Due to an increased interest in nonmetallic inclusions, a new subcommittee, designated Subcommittee IX, has been formed which will endeavor to prepare standard methods of determining inclusions. This new subcommittee is under the chairmanship of Samuel Epstein.

Because of lack of an immediate objective Subcommittee II on Definitions and Nomenclature has been discharged.

Committee E-4 reports a net gain in membership during the year of two members, bringing the total membership to 57.

TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The committee recommends that the following tentative standards for which the committee is responsible be continued as tentative without revision:

Tentative Methods of Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (E 2 – 39 T),

Tentative Methods of Preparation of Metallographic Specimens (E 3 – 39 T), and

Tentative Classification of Austenite Grain Size in Steels (E 19 – 39 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (E. H. Dix, Jr., chairman), which is responsible for the preparation of the Tentative Methods of Preparation of Metallographic Specimens (E 3 – 39 T), is sponsoring the round-table discussion on lapping methods and electrolytic polishing, mentioned above, in order to ascertain whether the status of these procedures would warrant incorporating them in Tentative Methods E 3 – 39 T.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee E-4 reported the following results of the letter ballot vote of a total of 38 ballots, returned from a committee membership of 53: E 2 - 39 T, affirmative 38, negative 0; E 19 - 39 T, affirmative 34, negative 1, ballots marked "not voting" 3.

[2] 1939 Book of A.S.T.M. Standards, Part I, pp. 1214, 1175.

Subcommittee VI on X-ray methods (W. L. Fink, chairman) held a meeting during the Metal Congress in Chicago, in October, 1939, at which the subject of diffraction methods was discussed. This subcommittee is attempting to establish a center of information on diffraction methods of chemical analysis.

Subcommittee VII on Recommended Practice for Dilatometric Analysis (L. L. Wyman, chairman) is reviewing the results of various recommendations with respect to methods and precision in dilatometric procedures as used in a number of laboratories, with the view of establishing a recommended practice in this field.

Subcommittee VIII on Grain Size (M. A. Grossmann, chairman) accomplished last year the revision of the metallographic grain size reference standards, which are now published under the title Tentative Classification of Austenite Grain Size in Steels (E 19 – 39 T).

Due to the widespread interest shown at the last meeting of Committee E-4, this subcommittee has been requested to investigate the feasibility of establishing a ferrite grain size standard.

The present officers of the committee were re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 57 members; 44 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

J. J. BOWMAN,
Secretary.

REPORT OF COMMITTEE E-7

ON

RADIOGRAPHIC TESTING

Committee E-7 on Radiographic Testing met at Atlantic City on June 26, 1939, and will hold a further meeting during this annual meeting. The committee has been active through its several subcommittees as indicated briefly below.

Subcommittee I on Radiography of Cast Metal (C. W. Briggs, chairman).—At the meeting of Committee E-7 in June, 1939, Subcommittee I reported a recommendation to adopt the U. S. Navy Radiographic Standards (Gamma-Ray) for pressure castings. The committee, after discussion, accepted this recommendation and it was subsequently approved by a letter ballot vote of the committee. The standards are to be presented subsequent to this annual meeting to Committee E-10 on Standards[1] for consideration as A.S.T.M. tentative standard.

The question of X-ray radiographic standards for steel castings to supplement those covering the gamma-ray field was discussed at the meeting in June, 1940. It was decided that such standards should be prepared and an attempt made to provide graded conditions of soundness. It is intended to have different standards applicable to different types of castings, and in many cases to different parts of the same casting. A special committee was appointed, with C. W. Briggs as chairman, to prepare the standards.

Subcommittee I has considered the question of revision of the Tentative Methods of Radiographic Testing of Metal Castings (E 15 - 39 T). After discussion in the subcommittee and at the meeting of the main committee, it was decided to provide for revision of this procedure in a comprehensive document in which radiographic procedures for both X-rays and gamma-rays are to be detailed and brought completely up to date. It is expected that two years will be required to complete the work. The committee in charge is headed by J. J. Curran.

Subcommittee II on Research and Development (H. E. Seemann, chairman).—During the year the very important paper by Lauriston S. Taylor on "Industrial X-Ray Protection"[2] was made available to various industrial plants where protection is an important problem.

Papers on "Precision Radiography," by G. E. Doan, and on "Some Aspects of Radiographic Sensitivity in Testing with X-Rays," by H. H. Lester were also published last year.[3]

The committee sponsored six papers being presented at this annual meeting of the Society. Three of these are to be given at a special session devoted to a "Topical Discussion on Radiographic Testing of Airplane Components,"[4] as follows:

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[1] The standard was considered by Committee E-10 but was returned for certain revisions. It is expected that the revised standard will be ready for resubmission shortly.

[2] Published in ASTM BULLETIN, No. 99, August, 1939, p. 23.

[3] Published in ASTM BULLETIN, No. 97, March, 1939, p. 27; No. 100, October, 1939, p. 33.

[4] To be published in ASTM BULLETIN.

"Quality and Quantity X-Ray Examination of Aircraft Material," by T. A. Triplett.
"Radiography of Aluminum Castings and Forgings for Airplanes," by Kent Van Horn.
"Technique and Organization in the Radiography of Aircraft Castings," by L. W. Ball.

The other three papers will be presented at the second session of the annual meeting, and are as follows:

"The Technique of Inspecting Wood Poles by X-Rays," by Myron Zuckor.[5]
"The Reduction of Secondary Radiation and of Excessive Radiographic Contrast by Filtration," by H. E. Seemann.[6]
"Precision Radiography-II," by Gilbert E. Doan and Winston H. Sharp.[7]

Following the meeting on radiographic inspection of aircraft material, a special committee was provided for under the chairmanship of A. W. F. Green, to consider and report on the question of standardized procedures for radiographic examination of airplane material.

Mention should be made of the remarkably fine radiographic display presented at the annual meeting. The exhibition committee was headed by Victor Hicks. The Westinghouse X-Ray Co. contributed generously in the loan of display cabinets.

Respectfully submitted on behalf of the committee,

H. H. LESTER,
Chairman.

R. A. GEZELIUS,
Secretary.

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[5] Published in ASTM BULLETIN, No. 106, October, 1940, p. 19.
[6] See p. 1289.
[7] See p. 1301.

REPORT OF COMMITTEE E-8

ON

NOMENCLATURE AND DEFINITIONS

Since the previous report to the Society in 1934, Committee E-8 on Nomenclature and Definitions has held one meeting: in New York, N. Y., on May 22, 1940. Two meetings of the Advisory Committee have been held: both in New York, N. Y., one on June 28, 1937, and the other on May 22, 1940. In recent years there have been relatively few definitions requiring final action on the part of Committee E-8 and accordingly it has not been necessary for the committee to meet annually as had been the custom. As a result of the considerations given by the committee at its recent meetings, action has been taken on several recommendations for the adoption of certain definitions as standard as well as the initiation of other subjects dealing with nomenclature and definitions of general interest to the standing committees of the Society.

The Advisory Committee is now functioning on a rotational basis with the terms of two members expiring each year. P. V. Faragher and L. H. Fry were appointed by the Executive Committee of the Society for a three-year term, expiring in 1942.

An increasing interest in the establishment of authoritative nomenclature and definitions has been evidenced on the part of the standing committees of the Society. This is particularly true in the case of new committees organized during the past several years. The committee has received a discussion by H. C. Porter proposing that consideration be given to the preparation of a recommended form to serve as a basis for the establishment of a definition, and when a term has several meanings, to provide a means of indicating the primary and secondary importance of the subjects covered.

A statement prepared by J. Spotts McDowell has been submitted to the committee by M. E. Holmes, the representative of Committee C-8 on Refractories. This statement also points out the need for the formulation of adequate definitions which contain in concise form that which is necessary to identify the materials or terms defined. It was recognized that the setting up of guiding principles for developing definitions would be of distinct value to the standing committees of the Society. It was accordingly decided by the Advisory Committee at its last meeting to publish an article along these lines in the ASTM BULLETIN for the purpose of eliciting discussion. It was thought that this method of presenting the subject to the Society membership would serve to bring the matter to the attention not only of the standing committees but also of others interested.

The Standard Definitions of Terms Relating to Heat Treatment Operations (Especially as Related to Ferrous Alloys) (A 119 – 33) were placed under the jurisdiction of Committee E-8 in 1936 when the former Committee A-4 on

Heat Treatment of Iron and Steel was discontinued. These definitions were prepared originally by the Joint Committee consisting of representatives of the American Society for Metals, Society of Automotive Engineers, and the A.S.T.M. It appeared desirable to give consideration to a revision of this standard in view of certain developments that had taken place since its preparation by the Joint Committee and adoption by the three societies. The present definitions apply only to the ferrous metals but similar terminology concerning heat treatment operations is also of interest in the field of non-ferrous metals. An A.S.M. glossary of terms, issued in 1939,[1] includes certain new definitions as well as revisions of those prepared by the Joint Committee. It was accordingly decided to appoint a special subcommittee to study the matter and to make recommendations as to cooperation on this subject with the other interested societies as well as on the problem of revising and enlarging the definitions.

Certain of the terms defined in the Standard Definitions of Terms Relating to Specific Gravity (E 12 – 27) have recently been commented on and in view of the suggestions received it has been decided to have the standard reviewed, especially since no change has been made in the definitions since they were adopted 13 years ago. A special subcommittee, composed of representatives of all interested standing committees, is to be appointed to review these definitions.

The committee has reviewed the Tentative Definitions of Terms Relating to Gypsum (C 11 – 31 T) which Committee C-11 is prepared to recommend for adoption as standard. Questions were raised concerning several of the definitions and these will be discussed with Committee C-11 during the year.

There have been recent indications of a need for a more closely defined distinction between the meaning of the terms "mechanical properties" and "physical properties." It would appear that these terms are quite often used interchangeably, and since the former is in general considered to have a more restrictive meaning it was thought desirable to outline more clearly the limitations of these two terms. Such a discussion would be useful as a guide in the preparation of specifications as well as to the authors of technical papers. In view of the general interest in this subject an article discussing the meaning and significance of these terms in the testing of engineering materials is to be prepared by L. H. Fry for publication in the ASTM BULLETIN.

The committee has received several requests for a definition of "baked enamel." This request has been referred for consideration to the representative of Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

Glossary of Terms.—The republication of a glossary of the terms appearing in the standards and tentative standards of the Society was again deferred last year partly as an economy measure but primarily because it appeared desirable to delay the preparation of a current list until after the publication of the 1939 Book of Standards. Consideration will be given this year to the preparation of an up-to-date glossary.

APPROVAL OF DEFINITIONS

The committee has also considered recommendations of several standing committees for the adoption as standard of definitions prepared by these committees, which recommendations appear in the annual reports being submitted to the Society at this annual meeting, as discussed below.

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[1] Glossary of Selected Iron and Steel Terms and Definitions of Terms Relating to Heat Treatment Operations, *Metals Handbook*, Am. Soc. for Metals, 1939 Edition, p. 3.

Revisions in the Standard Definitions of Terms Relating to Paint (D 16 – 24) are being recommended for immediate adoption by Committee D-1.[2] Certain suggestions of an editorial nature have been made by Committee E-8 concerning several of the definitions and these are being discussed with Committee D-1. The definitions, subject to acceptance by Committee D-1 of the editorial suggestions, have been submitted to letter ballot of Committee E-8 which consists of 48 members; 45 members returned their ballots, of whom 27 voted affirmatively, 4 negatively, and 14 marked their ballots "not voting."

Committee D-5 on Coal and Coke is recommending the adoption as standard of the Tentative Definition of the Term Coke (D 121 – 26 T), as revised.[3] The revised definition has been considered and approved by the Subcommittee on Coke, as mentioned later in this report. It has also been referred to letter ballot of Committee E-8 which consists of 48 members; 45 members returned their ballots, of whom 33 voted affirmatively, 4 negatively, and 8 marked their ballots "not voting."

The Tentative Definitions of Terms Relating to Textile Materials (D 123 – 39 T), which are being recommended by Committee D-13 for adoption as standard this year,[4] have been reviewed by Committee E-8. As a result of this review the definitions of certain terms have been questioned, and attention has been called to the desirability of including only those terms relating to textile materials not likely to be understood by the layman unless defined. Also, certain general terms have been defined, such as water vapor pressure, absolute and relative humidity, gage, tensile strength, etc., and Committee E-8 is of the opinion that particular attention should be given to such definitions to determine whether they are essential, and if so, whether they are correctly defined since they may have both a general and specific application. Committee E-8 has accordingly recommended that Committee D-13 defer the adoption as standard of these definitions so that they may be reviewed along the lines indicated and any changes or improvements incorporated before they are formally adopted by the Society as standard.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee on Definitions of Sand and Aggregate (Cloyd M. Chapman, chairman).—The Tentative Definition of the Term Aggregate (C 58 – 28 T) which has been continued in the tentative status for the past 12 years has not been recommended for adoption as standard since final agreement has not been reached on the definition by the several committees concerned. In order to harmonize the viewpoints that have been expressed, the following revised definition is now being submitted to this subcommittee:

Aggregate.—Inert material, such as sand, gravel, slag, or broken stone, or combinations thereof, used in combination with a cementing medium to form a conglomerated mass such as mortar, plaster, concrete, mastic, etc.

Subcommittee on Definition of Sieve and Screen (Cloyd M. Chapman, chairman).—This subcommittee has had under consideration the Tentative Definition of the Term Screen (Sieve) (E 13 – 28 T) which has also been continued without change for the past 12 years.

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[2] This recommendation was withdrawn and in view of additional revisions Committee D-1 recommended that the revised Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products be published as tentative, see p. 282.

[3] In deference to a suggestion from the Executive Committee of the Society that this definition be not adopted, Committee D-5 voted at its meeting on June 27, 1940, not to present the revised definition, see Editorial Note, p. 340.

[4] In presenting the definitions to the Society for adoption as standard Committee D-13 incorporated certain of the suggestions as editorial changes. The definitions as adopted appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 143.

The subcommittee reviewed this definition by letter in 1938 as a result of which it was decided to change the word "rectangular" in Note 2 to read "square." With this change, the definition will read as follows:

> *Screen (Sieve).*—A plate or sheet or a woven cloth, or other device, with regularly spaced apertures of uniform size, mounted in a suitable frame or holder, for use in separating material according to size.
>
> NOTE 1.—The shape and spacing of apertures, size of wires or threads, thickness of plate or sheet, allowable variations and similar properties should be taken care of in specifications.
>
> NOTE 2.—In mechanical analysis testing work, when not otherwise specified, the term "sieve" shall apply to an apparatus in which the apertures are square, and the term "screen" shall apply to an apparatus in which the apertures are circular.

The above revised definition is now being considered for adoption as standard.

Subcommittee on Definition of Concrete (Cloyd M. Chapman, chairman).—This subcommittee has under consideration the formulation of a definition for "concrete." A proposed definition which appeared in the 1928 report[5] as information, for the purpose of eliciting comments and suggestions, is to be further reviewed and discussed with interested committees of the Society. In this connection consideration will also be given to the definitions for "gypsum concrete" and "gypsum fiber concrete" prepared by Committee C-11 on Gypsum and which are now considered by that committee to be suitable for adoption as standard.

Subcommittee on Definitions Relating to Bituminous Materials (K. G. Mackenzie, chairman).—This subcommittee has been studying the two existing definitions for water-gas tar prepared, respectively, by Committees D-4 on Road and Paving Materials and D-7 on Timber. The following revised definition proposed by W. H. Fulweiler, a member of the subcommittee, is now being discussed and it is hoped that the committee may reach agreement on a recommended definition to be submitted for adoption as standard to replace the two present definitions of this term:

> *Water-Gas Tar.*—A tar composed essentially of aromatic hydrocarbons produced in the manufacture of carbureted water gas by the transformation at high temperatures of petroleum oil in the presence of blue gas.

Subcommittee on Definition of Coke (H. C. Porter, chairman).—It is recommended by this subcommittee that the Tentative Definition of the Term Coke (D 121 - 26 T) be revised[3] as follows:

Change the definition of the term coke from its present form: namely,

> *Coke.*—The infusible, cellular, coherent solid material obtained from coal as the residue from its destructive distillation.
>
> NOTE 1.—Such material obtained from the destructive distillation of petroleum or petroleum residues is known as "petroleum coke."
>
> NOTE 2.—Such material obtained from the destructive distillation of coal tar pitch is known as "pitch coke."

to read as follows:

> *Coke*: (*a*) The solid, cellular, coherent residue from destructive distillation of bituminous coal, said residue having such physical structure as results from the hardening of a fused or semiliquid mass.
>
> (*b*) If used with appropriate qualifying adjective, a similar residue from petroleum, pitch, and some other carbonaceous materials.

The revised definition has been submitted to letter ballot of this subcommittee and has received the unanimous approval of that group, which consists of one representative from each of the following committees: A-3 on Cast Iron, D-2 on Petroleum Products and Lubricants, D-4 on Road and Paving Materials, and D-5 on Coal and Coke.

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 679 (1928).

Subcommittee on Definitions of Net and Gross Calorific Values of Fuels (H. C. Porter, chairman).—This subcommittee has given consideration to the choice of a single term to indicate "calorific value" or "heat of combustion" in view of the inconsistent use at the present time of the several terms "calorific value," "thermal value," "heat of combustion," and "heating value." This matter was referred by letter ballot to the members of the subcommittee which has resulted in a majority of the members favoring the term "calorific value." Several members of the committee favored the use of the term "heat of combustion" as perhaps more accurately descriptive, since it narrows precisely the application of that heat which is produced by combustion. The term "calorific value" was favored by the majority since it appeared to have the advantage of already being in more general use, having been adopted by the British Standards Institution and being similar to the use of "pouvoir calorique" by the French standardization society and to "Heizwert" in German standards. In view of the preference that has been expressed for both these terms, the possibility of using a combined term "calorific value (heat of combustion)" has been suggested. The committee would be interested in receiving comments on this matter from members of other standing committees and from the Society membership.

In connection with the Tentative Definitions of Terms Gross Calorific Value and Net Calorific Value of Fuels (D 407 – 35 T), the chairman of the subcommittee proposed the alternative or optional use of the following terms as a result of suggestions and opinions received from authoritative outside sources: (*1*) gross calorific value, and net calorific value or (*2*) higher heat of combustion, and lower heat of combustion. This suggestion was considered by Committee E-8 and the committee has recommended that the subject be given further study by the subcommittee.

Cloyd M. Chapman was re-elected chairman of the committee for the ensuing term of two years.

This report has been submitted to letter ballot of the committee which consists of 48 members; 47 members returned their ballots, of whom 45 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

CLOYD M. CHAPMAN,
Chairman.

R. E. HESS,
Ex-officio Secretary.

REPORT OF COMMITTEE E-9
ON
RESEARCH

With this report Committee E-9 on Research reviews briefly certain research activities and matters related thereto which have engaged its attention during the past year. As customary, a more comprehensive review of research activities, with particular reference to such activities in the standing committees which are only mentioned herein, will appear in the October ASTM BULLETIN.

Research Committee Activities:

The *Research Committee on Fatigue of Metals* has nearly completed the investigation of effect of type of testing machine on results of fatigue tests as originally planned.[1] In considering the data so far obtained, the committee concluded that additional tests would be necessary, because of the amount of "scatter" in certain of the fatigue test results and the advisability of including more tests at high stresses to study the shape of the upper part of the *S–N* diagrams. Committee E-9 has contributed $200 from the Society's Research Fund towards this extension of the project, supplementing the $500 contributed a year ago. It is expected that an oral presentation of the results so far obtained will be given at this annual meeting.

The *Joint Research Committee on Effect of Temperature on the Properties of Metals* of the American Society of Mechanical Engineers and this Society is presenting a report[2] at this meeting of the Society describing its activities for the year. Projects covered in its report include those dealing with tubular members subjected to internal pressures, properties of metals at low temperatures, bolting performance at high temperature, listing of high-temperature testing equipment and methods, and correlation of data on K-20 steel. A second report dealing with the effect of manufacturing variables on the high-temperature properties of metals, one of the sponsored projects being carried out at Battelle Memorial Institute, is presented. Another project committee prepared an introduction to the subject of grain size of steel, in the form of a report by J. R. Vilella which has been published in *Mechanical Engineering*, April, 1940.

The committee is planning a series of cooperative tests for studying some of the important properties of copper-base and nickel-base alloys at high temperatures. Other projects in the planning stage relate to carbon-molybdenum steel and to the evaluation of a number of "short-time" test methods in terms of their ability to forecast long-time (creep) test results.

The *Joint Committee on Exposure Tests of Plating on Non-Ferrous Metals*, composed of representatives of the American Electroplaters' Society, the National Bureau of Standards and this Society, has continued its work during the past year, which has included the drafting of specifications for electroplated coatings in cooperation with appropriate committees of the Society. The exposure tests which are nearing completion were summarized in a paper by Blum and Strausser.[3] An informal report will be presented by the joint committee at this meeting.

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[1] See p. 120.
[2] See p. 121.

Research Progress of A.S.T.M. Committees:

Effect of Speed of Testing on Physical Properties of Metals (Committee E-1).—This investigation, sponsored by the Section on Effect of Speed of Testing, of Committee E-1, and carried out at the University of Illinois under an agreement between the University and the Society, has been completed, and the results are reported in a paper for this annual meeting by Paul G. Jones and H. F. Moore entitled "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals."[4] The paper describes results of tension tests of fourteen metals using different speeds of testing. The apparatus used was autographic and employed duplicate devices for weighing load; stretch of specimen was measured by means of an electric resistance extensometer. Time records were made photographically on an oscillograph film by contacts fitted to an electric clock. Extensive data showing influence of rate of strain on yield strength, tensile strength, elongation and reduction of area are presented, together with proposed methods for determining rates of strain using a type of testing machine in common use, not equipped with any special control apparatus. Committee E-9 contributed $300 from the Research Fund this year towards the completion of this project.

Testing of Textile Finishes (Committee D-13).—This project, which has for its objective the development of physical testing methods for the evaluation of various types of textile finishes, is being carried out by a research associate at the National Bureau of Standards and is supported financially by some ten industrial companies. A report of progress in the first year's work in studying such factors as limpness, smoothness and fullness of fabric, appears in the current report of Committee D-13 on Textiles.[5] Most of the present subscribers to the investigation have agreed to continue their support for another year and additional sponsors have been obtained.

Moisture Content of Paperboard and of Corrugated Fiberboard Containers (Committee D-6).—This project has for its primary purpose the securing of data on the moisture content of container grades of paperboard, and of corrugated fiberboard containers, resulting from conditioning these products in accordance with several specified procedures; also, data on the relationship between moisture content and certain physical properties. Six laboratories experienced in this type of testing cooperated under the direction of Subcommittee IV of Committee D-6 on Paper and Paper Products.

Effect of Pretreatments in Painting of Structural Steel (Committee D-1).—This project, being carried out under the direction of Subcommittee XXIX of Committee D-1 on Paint, Varnish, Lacquer, and Related Products, is designed to give information on the value of various types of inhibitive solutions applied to steel surfaces before painting. A number of steel panels have been given various pretreatments, painted and exposed to the atmosphere. Brief mention of the project is made in the current report of Committee D-1.[6]

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[3] William Blum and P. W. C. Strausser on "Outdoor Exposure Tests of Electroplated Nickel and Chromium Coatings on Steel and Nonferrous Metals," *Journal of Research*, Nat. Bureau Standards, Vol. 24, p. 443 (1940) (*Research Paper 1293*).

[4] See p. 610.

[5] See p. 411.

[6] See p. 289.

Committee E-9 has given $75 from the Research Fund to defray expenses incurred in the installation of the test panels.

Determination of Water Vapor in Gaseous Fuels (Committee D-3).—Through the School of Mineral Industries of Pennsylvania State College, under the direction of Prof. A. W. Gauger, Committee D-3 is engaged in the development of methods for determining water vapor in gaseous fuels—a determination which is of much importance to the fuel industries. The immediate need is for the establishment of a reference standard for the quantitative, absolute measurement of water vapor in air and fuel gases, and Committee E-9 has given $200 from the Research Fund to aid in the procurement of needed apparatus and thus supplement the extensive contributions of the college to this project in its various ramifications.

Capping Tests of Hollow Tile (Committee C-15).—The work on these tests, carried out at Rensselaer Polytechnic Institute, has been substantially completed and a report is being prepared for Committee C-15.

New Activities.—In addition to the projects just described, research activities on the following subjects have been started during the year by the committees named. Brief statements of each appeared in the ASTM BULLETIN for October, 1939:

Magnetic Properties under Conditions of Combined Alternating and Direct Magnetization—of chief interest in connection with core materials (Committee A-6).

Study of Pictorial Scale for Evaluating Rust Resistance (Committee D-1).[7]

Study of Methods of Testing Automotive Rubber—directly related to the work of the joint S.A.E.-A.S.T.M. committee (Committee D-11).

Rubber Life Tests—air pressure tests, kinetic aging tests under artificial light and interpretation of test data (Committee D-11).

Special mention should be made of the completion of an extensive research by Committee B-3 on the subject of Galvanic and Electrolytic Corrosion. The final report, which appears in the *Proceedings*[8] for 1939, gives the results of tests after seven years exposure in nine test locations representing seacoast, industrial and rural atmospheres. All couple combinations of the following metals were included in these tests: aluminum, copper, iron, lead, nickel, tin, and zinc. Data from the thousands of tests are given in tabular form with discussion by the committee in charge and will be of great value to all who are concerned with this problem.

Committee B-6 is about to undertake an extension of its program of research on die-cast metals and alloys, for which purpose it is planning to raise about $2000 from producers and consumers of these materials.

Research on Standardization of pH Measurements:

Committee E-9 has given careful consideration to the part that this Society might appropriately take in the proposed research on the standardization of pH measurements which has been suggested to be carried out under the general auspices of the National Bureau of Standards. A questionnaire to ascertain the extent of interest of the Society in this subject was circulated among all standing committees. Four committees expressed direct interest in the proposed research, namely, Committees D-6 on Paper and Paper Products, D-12 on Soap and Other Detergents, D-13 on Textile Materials, and D-19 on Water for Engineering Purposes; doubtless other committees would have some interest in having the Society develop standard methods for making pH determinations, or more broadly, hydrogen ion determinations.

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[7] See Proposed Reference Standards of Rusting of Painted Iron or Steel Surfaces, ASTM BULLETIN, No. 107, December, 1940, p. 25.

[8] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 247 (1939).

Committee E-9 has concluded that the Society is not in a position at this time to lend financial support to this research project. However, there seems definitely to be a need for development of standard methods for hydrogen ion determination in certain fields in which the Society's committees are engaged and Committee E-9 feels that A.S.T.M. might be able to make an effective contribution to the subject through the organization of a committee charged with the development of such methods. Such a committee could focus attention on the subject in the Society and would be in an excellent position to determine the degree of interest that the Society should properly have in any researches on this subject.

Accordingly, Committee E-9 recommended to the Executive Committee of the Society that a Committee on Hydrogen Ion Determinations be formed, either as a separate standing committee or as a subdivision of Committee E-1 on Methods of Testing as may appear desirable. Committee E-9 understands that Committee E-1 has recently agreed to form a Technical Committee of E-1 on this subject.

Proposed Research on Freezing-and-Thawing Tests:

During the year the Executive Committee referred to Committee E-9 a proposal that had developed from its consideration of a Round-Table Discussion on Freezing-and-Thawing Tests at the 1939 annual meeting, that there may be a need for a research committee to study the fundamental aspects of the phenomena of freezing and thawing of masonry and related materials. Consideration by Committee E-9 indicated the desirability of approaching the subject from the viewpoint of durability of such products in general and of conferring with other organizations that would have an important interest in research in this field, especially the Highway Research Board of the National Research Council, which has committees studying durability, and the American Concrete Institute. These organizations joined with this Society in holding a conference in Washington on May 27, 1940, attended by the leading investigators of the country in this field, at which it was decided to recommend that research in durability of concrete be developed under the guidance of a joint committee to be established by the three bodies named. Further consideration will be given to this subject by Committee E-9 during the annual meeting.

Research Activities of Other Societies:

National Research Council.—The Society is represented on the Division of Engineering and Industrial Research of this Council by L. M. Currie, and is also a member of the Highway Research Board of that Division, being represented by Prévost Hubbard with F. E. Richart as alternate. The annual *Proceedings* of the Highway Research Board covering its annual meeting in December, 1939, contain many reports and papers in the Department of Materials and Construction that are of interest to this Society and that come quickly to the attention of appropriate A.S.T.M. committees through interlocking memberships.

A.S.M.E. Research Committee on Mechanical Springs.—The Society is represented on this committee by C. T. Edgerton, chairman of the subcommittee on spring steel of Committee A-1. The committee is making slow but steady progress in the analysis and solution of various problems relating to helical spring design and is planning a research program on leaf springs. Reference is made to the following two papers presented at the annual meeting of the American Society of Mechanical Engineers, December, 1939: "Helix Warping in Helical Compression Springs," by D. H. Pletta and F. J. Maher, and "Calculation of the Elastic Curve of a Helical Compression Spring," by H. C. Keysor.

Engineering Foundation—Welding Research Committee.—The Society is represented on the Industrial Research Division of this committee by N. L. Mochel. Reference is made to a description of the work of this group in the December, 1939, issue of ASTM BULLETIN by its chairman, Col. G. F. Jenks. One specific project of direct interest to this Society is a study of the effect of carbon and manganese on the weldability of carbon steels. This project contemplates ultimately the study of 20 compositions of steel comprising five manganese ranges for each of four carbon ranges; a preliminary investigation will be carried out on the use of three steels of relatively low, medium, and relatively high combinations of carbon and manganese, for the purpose of developing if possible a standard test for relative weldability of steels, before undertaking so comprehensive an investigation. A test for weldability is of particular importance to Committee A-1 on Steel in connection with the development of specifications for steels to be welded. The Industrial Research Division is raising a fund of $9000 to support this project, of which the majority will be contributed by the iron and steel industry and by steel fabricators. Because of the importance of the work to this Society, Committee E-9 will contribute $250 from the Research Fund towards this project.

American Coordinating Committee on Corrosion:

The formation of this committee as a clearing house for information on corrosion work was announced in the report of Committee E-9 a year ago, and the March, 1939, issue of ASTM BULLETIN carried an account of its organization and the work that is being planned. During the year the committee completed its organization with the election of permanent officers: F. N. Speller, chairman; R. M. Burns, vice-chairman; and George H. Young, secretary. Compilation of a directory of research workers is nearing completion. A meeting of the committee is to be held during this annual meeting.

Fundamental Research in the Society:

Committee E-9 has considered the points developed in Dr. H. H. Lester's interesting letter in the January, 1938, ASTM BULLETIN entitled "A.S.T.M. in Basic Research," together with replies from various members of the Society published in the March and May BULLETINS for that year. There are obviously a number of aspects to the subject and these have been rather fully discussed. It is the consensus of the committee that the distinction between "basic" and "applied" research is often difficult to define and that the Society is properly interested in any phase of research whether basic or applied that will promote knowledge of materials, this being one of the purposes for which the Society is organized. In the words of one member, the Society is "vitally interested in the facts underlying tests and specifications, no matter how deep it has to dig for them. But it should stick to that field for there is plenty to do in it." On the whole, Committee E-9 feels that the research activities of the Society as at present carried on are meeting the needs developed through the activities of the various committees, within the limitations imposed by the financial resources of the Society.

Administrative Matters:

With this meeting the term of J. S. Miller expires and Arthur W. Carpenter, Manager of Testing Laboratories, The B. F. Goodrich Co., has been appointed for the term of five years.

The principal of the A.S.T.M. Research Fund is now $27,491.15. Since the report a year ago the principal was augmented by $1250 from the entrance fees for 1939, $200 from current (1940) entrance fees, $3000 from entrance fees prior to 1937 that had been temporarily withheld from the Fund, and a small item of $8.47 profit on maturity of a bond.

The balance of income in the Research Fund on May 31, 1940, was $824.23. This is after payment of the $775 covering the projects on fatigue of metals, effect of speed of testing, pretreatment in painting of steel, and determination of water vapor in gaseous fuels mentioned heretofore, but before payment of $250 appropriated to the Welding Research Committee of Engineering Foundation. The present annual available income from the principal of the Fund is about $950.

Respectfully submitted on behalf of the committee,

T. S. FULLER,
Chairman.

C. L. WARWICK,
Secretary.

REPORT OF COMMITTEE E-10

ON

STANDARDS

Committee E-10 on Standards has had a fairly active year. Two meetings of the committee were held: in Philadelphia, Pa., on August 24, 1939, and in New York, N. Y., on January 19, 1940.

The membership on Committee E-10 of Cloyd M. Chapman and H. L. Curtis expired last year. The committee wishes to record its appreciation of the helpful and unstinted services of these men, Mr. Chapman having been a member of the committee since its organization in 1929. The Executive Committee appointed F. E. Richart of the University of Illinois, and H. S. Vassar of the Public Service Electric and Gas Co. to serve as members of Committee E-10 for a three-year term, expiring in 1942.

R. P. Anderson was elected chairman of the committee to fill Mr. Chapman's unexpired term (until June, 1940).

Jurisdiction over the Regulations Governing the Form but not the Substance of Standards, formerly under Committee E-5 on Standing Committees, was transferred to Committee E-10, and the Regulations were revised during the year. They had stood without revision since 1921 and since certain minor deviations had been found desirable in the arrangement of standards, the current practice and the Regulations were not entirely in accord. The changes made in the Regulations were for the purpose of converting them into a guide or set of recommendations rather than a form to be rigidly adhered to; the title of the Regulations was accordingly changed to Recommendations on the Form of Standards. The Recommendations as revised were printed in the 1939 Year Book of the Society.

As in the previous year, a very considerable number of new tentative standards and revisions of existing standards and tentative standards have been submitted to Committee E-10 for approval, as given in detail later in this report. For the most part these were considered at the August meeting of the committee.

REVIEW OF A.S.T.M. STANDARDIZATION ACTIVITIES

A general summary of the new and revised tentative standards submitted at the 1939 annual meeting was included in the August issue of the ASTM BULLETIN together with a review of the standardization work of the various committees, with statements concerning new standards in prospect. Much of the information, particularly with respect to standardization projects under development in committees, is furnished by the standing committees subsequent to the annual meeting and takes advantage of the fact that many committees plan their next year's work at committee meetings held during the annual meeting.

At the present writing, it is possible to make only a preliminary estimate in terms of new and revised standards of the volume of standardization work accomplished during the year and reported to the Society at this meeting. The reports of standing committees to be presented at the annual meeting include recommendations to the Society substantially as follows:

New tentative standards	77
Revisions of existing tentative standards	68
Tentative revisions of existing standards	22
Tentative standards recommended for adoption as standard	34
Revisions of existing standards recommended for adoption as standard	67
Standards and tentative standards withdrawn or replaced	18

There are at present 561 standards and 324 tentative standards of the Society, making a total of 885, and if the above recommendations are approved this total will become 944.

CONSIDERATION OF NEW AND REVISED STANDARDS

Proposed new standards approved by Committee E-10 and published as tentative during the year are as follows:

Tentative Specifications for:

- Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 39 T), submitted by Committee A-1 on Steel,
- Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 39 T), submitted by Committee A-1,
- Hard-Drawn Steel Spring Wire (A 227 – 39 T), submitted by Committee A-1,
- Steel Music Spring Wire (A 228 – 39 T), submitted by Committee A-1,
- Oil-Tempered Steel Spring Wire (A 229 – 39 T), submitted by Committee A-1,
- Carbon-Steel Valve Spring Quality Wire (A 230 – 39 T), submitted by Committee A-1,
- Chromium-Vanadium Steel Spring Wire (A 231 – 39 T), submitted by Committee A-1,
- Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 – 39 T), submitted by Committee A-1,
- Iron and Steel Arc-Welding Electrodes (A 233 – 40 T), submitted by Committee A-1,
- Cast-Iron Pit-Cast Pipe for Water and Other Liquids (A 44 – 39 T), submitted by Committee A-3 on Cast Iron,
- Cast Iron Soil Pipe and Fittings (A 74 – 39 T), submitted by Committee A-3,
- Soft Rectangular and Square Copper Wire for Electrical Conductors (B 48 – 39 T), submitted by Committee B-1 on Copper and Copper Alloy Wires for Electrical Conductors,
- Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 – 39 T), submitted by Committee B-2 on Non-Ferrous Metals and Alloys,
- Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 39 T), submitted by Committee B-7 on Light Metals and Alloys,
- Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544 – 39 T), submitted by Committee D-4 on Road and Paving Materials,

Tentative Methods of:

- Chemical Analysis of Glass Sand (C 146 – 39 T), submitted by Committee C-14 on Glass and Glass Products,
- Hydrostatic Pressure Test on Glass Containers (C 147 – 39 T), submitted by Committee C-14,
- Polariscopic Examination of Glass Containers (C 148 – 39 T), submitted by Committee C-14,
- Thermal Shock Test on Glass Containers (C 149 – 39 T), submitted by Committee C-14,
- Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 – 39 T), submitted by Committee D-4 on Road and Paving Materials,
- Test for Sieve Analysis of Mineral Filler (D 546 – 39 T), submitted by Committee D-4,
- Test for Index of Dustiness of Coal and Coke (D 547 – 39 T), submitted by Committee D-5 on Coal and Coke,
- Test for Water-Soluble Acidity or Alkalinity of Paper (D 548 – 39 T), submitted by Committee D-6 on Paper and Paper Products,
- Test for Resin in Paper (D 549 – 39 T), submitted by Committee D-6,
- Testing Glass Spool Insulators (D 550 – 39 T), submitted by Committee D-9 on Electrical Insulating Materials,
- Measuring Shrinkage from Mold Dimensions of Molded Materials Used for Electrical Insulation (D 551 – 39 T), submitted by Committee D-9,

Testing Sponge Rubber Products (D 552 - 39 T), submitted by Committee D-11 on Rubber Products,

Test for Viscosity and Total Solids Content of Rubber Cements (D 553 - 39 T), submitted by Committee D-11,

Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (E 2 - 39 T), submitted by Committee E-4 on Metallography,

Radiographic Testing of Metal Castings (E 15 - 39 T), submitted by Committee E-7 on Radiographic Testing,

Tentative Classification of:

Austenite Grain Size in Steels (E 19 - 39 T), submitted by Committee E-4 on Metallography, and

Tentative Recommended Practice for:

Conducting Plant Corrosion Tests (A 224 - 39 T), submitted by Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys.

Revisions of the following tentative standards and standards were accepted by Committee E-10:

Tentative Specifications for:

Alloy-Steel Bolting Materials for High-Temperature Service from 750 to 1100 F., Metal Temperatures (A 193 - 39 T), submitted by Committee A-1 on Steel,

Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 39 T), submitted by Committee A-1,

Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 39 T), submitted by Committee A-1,

Brass Sheet and Strip (B 36 - 39 T), submitted by Committee B-5 on Copper and Copper Alloys,

Copper-Silicon Alloy Plate and Sheet (B 96 - 39 T), submitted by Committee B-5,

Leaded Brass Sheet and Strip (B 121 - 39 T), submitted by Committee B-5,

Leaded-Nickel-Brass and Leaded-Nickel-Bronze (Nickel-Silver) Alloys in Ingot Form for Sand Castings (B 123 - 39 T), submitted by Committee B-5,

Sewer Brick (Made from Clay or Shale) (C 32 - 39 T), submitted by Committee C-15 on Manufactured Masonry Units,

Building Brick (Made from Clay or Shale) (C 62 - 39 T), submitted by Committee C-15,

Fuel Oils (D 396 - 39 T), submitted by Committee D-2 on Petroleum Products and Lubricants,

Asphalt Filler for Brick Pavements (D 241 - 39 T), submitted by Committee D-4 on Road and Paving Materials,

Primer for Use with Asphalt in Dampproofing and Waterproofing (D 41 - 39 T), submitted by Committee D-8 on Bituminous Waterproofing and Roofing Materials,

Tentative Methods of:

Test for Dielectric Strength of Anodized Aluminum (B 110 - 39 T), submitted by Committee B-7 on Light Metals and Alloys,

Test for Loss on Heating of Oil and Asphaltic Compounds (D 6 - 39 T), submitted by Committee D-4 on Road and Paving Materials,

Testing Varnishes Used for Electrical Insulation (D 115 - 39 T), submitted by Committee D-9 on Electrical Insulating Materials,

Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 39 T), submitted by Committee D-9,

Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 39 T), submitted by Committee D-9,

Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 39 T), submitted by Committee D-9,

Testing Pin-Type, Lime Glass Insulators (D 468 - 39 T), submitted by Committee D-9,

Testing Hard Rubber Products (D 530 - 39 T), submitted by Committee D-11 on Rubber Products,

Standard Specifications for:

Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 37), submitted by Committee A-1 on Steel,

Carbon-Steel and Alloy-Steel Forgings (A 18 - 30), in the form of new Tentative Specifications for Carbon-Steel Forgings (A 18 - 39 T), submitted by Committee A-1,

Standard Methods of:

Sampling and Testing Brick (C 67 - 39), in the form of new Tentative Method of Freezing-and-Thawing Test of Brick (C 67 - 39 T), submitted by Committee C-15 on Manufactured Masonry Units,

Testing Molded Materials Used for Electrical Insulation (D 48 - 39), submitted by Committee D-9 on Electrical Insulating Materials,

- Test for Grading and Classification of Natural Mica (D 351 - 38), in the form of new Tentative Method of Test for Power Factor and Dielectric Constant of Natural Mica (D 351 - 39 T), submitted by Committee D-9, and
- Test for Rockwell Hardness of Metallic Materials (E 18 - 36), in the form of new Tentative Method of Test for Rockwell Superficial Hardness of Metallic Materials (E 18 - 39 T), submitted by Committee E-1 on Methods of Testing.

The withdrawal of the following standards and tentative standards was approved by Committee E-10:

Standard Specifications for:

Marine Boiler Steel Plates (A 114 - 33), recommended by Committee A-1 on Steel,

Standard Recommended Practice for:

Radiographic Testing of Metal Castings (E 15 - 29), recommended by Committee E-7 on Radiographic Testing,

Standard Rules:

Governing the Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (E 2 - 36), recommended by Committee E-4 on Metallography,

Standard Grain Size Chart:

for Classification of Steels (E 19 - 33), recommended by Committee E-4,

Tentative Specifications for:

Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods) (A 205 - 37 T), recommended by Committee A-1 on Steel,

Quicklime for Structural Purposes (C 5 - 34 T), recommended by Committee C-7 on Lime,

Hydrated Lime for Structural Purposes (C 6 - 34 T), recommended by Committee C-7, and

Tentative Grain Size Chart:

for Classification of Steels (E 19 - 38 T), recommended by Committee E-4 on Metallography.

PROMOTION AND EXPANSION OF STANDARDIZATION ACTIVITIES

One of the functions of Committee E-10 is to give consideration to new work that should be undertaken and fields in which there is definite demand that standards be developed. While several projects are now before the committee for consideration, the committee is not as yet prepared to make any definite recommendations concerning expansion of the Society's standardization work in the immediate future.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Standards Submitted Under Existing Standards and Proprietary Procedure.—The existing standard method for submitting standards to the American Standards Association, whereby it is necessary to show that the standard is supported by the necessary consensus of those substantially concerned with its scope and provisions, is particularly appropriate for a large number of A.S.T.M. standards. Under this procedure the following standards have been approved during the year by the A.S.A. as American standard:

Standard Specifications for:

Round Timber Piles (D 25 - 37; A.S.A 06—1939), and

Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (D 245 - 37; 07—1939).

The Society has been granted proprietary sponsorship for the revisions of standards approved under the existing standards method and, functioning as proprietary sponsor, it has submitted to the A.S.A. during the year revisions of the following standards, all of which have been approved:

Standard Specifications for:

Steel for Bridges and Buildings (A 7 - 39; G24—1939),

Mild Steel Plates (A 10 - 39; G20—1939),

Billet-Steel Bars for Concrete Reinforcement (A 15 - 39; A50.1—1939),

Alloy-Steel Bolting Material for High-Temperature Service (A 96 - 39; G17.2—1939),

Forged or Rolled Steel Pipe Flanges for High-Temperature Service (A 105 - 39; G17.3—1939),
Structural Rivet Steel (A 141 - 39; G21—1939),
Uncoated Wrought-Iron Sheets (A 162 - 39; G23—1939),
Copper Water Tube (B 88 - 39; H23.1—1939),
Gypsum Plasters (C 28 - 39; A49.3—1939),
Zinc Oxide (D 79 - 39; K22—1939),
Red Lead (D 83 - 39; K24—1939),
Prussian Blue (D 261 - 39; K29—1939),
Reduced Para Red (D 264 - 39; K31—1939),
Titanium Dioxide Pigments (D 476 - 39; K45—1939),

Tentative Specifications for:

Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 39 T; H7—1939), approved as American tentative standard,

Standard Methods of:

Chemical Analysis of White Pigments (D 34 - 39; K15—1939),
Test for Specific Gravity of Pigments (D 153 - 39; K41—1939), and
Testing Woven Textile Fabrics (D 39 - 39; L5—1939).

Standards Submitted Under Sectional Committee Procedure.—Reports were received from a number of the sectional committees for which the Society is sponsor, submitting standards to the A.S.A. for approval. These reports were transmitted to that association and the following standards have been approved during the year as American standard, with the exception of one which was approved as American tentative standard, as indicated:

On the Recommendation of Sectional Committee A21 on Cast-Iron Pipe and Fittings:

Specifications for Cast-Iron Pit-Cast Pipe for Water and Other Liquids (A 44 - 39 T; A.S.A. A21.2—1939).

On the Recommendation of Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing:

Specifications for Welded Wrought-Iron Pipe (A 72 - 39; B36.2—1939),
Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 39; B36.3—1939),
Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 - 39; B36.4—1939), and
Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 - 39; B36.9—1939).

On the Recommendation of Sectional Committee C59 on Electrical Insulating Materials:

Specifications for Rubber Insulating Tape (D 119 - 38; C59.6—1939),
Methods of Testing Molded Materials Used for Electrical Insulation (D 48 - 39; C59.1—1940), and
Methods of Test for Insulation Resistance of Electrical Insulating Materials (D 257 - 38; C59.3—1939).

On the Recommendation of Sectional Committee H4 on Copper Wire:

Specifications for Hard-Drawn Copper Wire (B 1 - 39; H4.2—1940),
Specifications for Medium-Hard-Drawn Copper Wire (B 2 - 39; H4.3—1940),
Specifications for Soft or Annealed Copper Wire (B 3 - 39; H4.1—1940),
Specifications for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 - 39; H4.4—1940),
Specifications for Bronze Trolley Wire (B 9 - 39; H4.5—1940),
Specifications for Copper Trolley Wire (B 47 - 39; H4.6—1940), and
Specifications for Hot-Rolled Copper Rods for Electrical Purposes (B 49 - 39; H4.7—1940).

On the Recommendation of Sectional Committee M20 on Classification of Coals:

Definitions for Commercial Varieties of Bituminous and Subbituminous Coals (D 493 - 39; M20.4—1939).

On the Recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants:

Method of Test for Cloud and Pour Points (D 97 - 39; Z11.5—1939),
Method of Test for Distillation of Natural Gasoline (D 216 - 39; Z11.11—1939),
Method of Test for Sulfur in Petroleum Oils by Bomb Method (D 129 - 39; Z11.13—1939),
Method of Test for Thermal Value of Fuel Oil (D 240 - 39; Z11.14—1939),
Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 39; Z11.25—1939),
Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287 - 39; Z11.31—1939),

- **Method of Test for Burning Quality of Kerosine Oils (D 187 - 39; Z11.17—1939),**
- **Method of Test for Knock Characteristics of Motor Fuels (D 357 - 39; Z11.37—1939),**
- **Viscosity-Temperature Charts for Liquid Petroleum Products (D 341 - 39; Z11.39—1939),**
- **Definitions of Terms Relating to Petroleum (D 288 - 39; Z11.28—1939), and**
- **Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (D 155 - 39 T; Z11.34—1939), approved as American tentative standard.**

The Method of Test for Expressible Oil and Moisture in Paraffin Waxes (D 308 - 29 T; Z11.27—1932) which had been approved as American tentative standard was withdrawn.

Sectional Committee M20 on Classification of Coals.—During the year Sectional Committee M20, for which the Society was sponsor, completed its work and was discharged. The work of this committee was initiated in 1926. Following several years of active research the committee's work led to the approval of four standards which represented the completion of its assignment. With the discharge of the committee, the Society has been granted proprietary sponsorship for future revisions of the four standards, which have been placed under the jurisdiction of Committee D-5 on Coal and Coke. Arrangements have been made for representation on Committee D-5 of any organizations formerly on the sectional committee.

Respectfully submitted on behalf of the committee,

R. P. ANDERSON,
Chairman.

C. L. WARWICK,
Ex-officio Secretary.

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

The purpose of the Edgar Marburg Lecture is to have described at the annual meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first secretary of the Society.

PORTLAND CEMENT

THEORIES (PROVEN AND OTHERWISE) AND SPECIFICATIONS

FIFTEENTH EDGAR MARBURG LECTURE[1]

BY P. H. BATES[2]

Definition of Portland Cement:

If it were possible tersely and adequately to define portland cement, the development of the subject matter of this lecture would follow quite different lines from those which will be pursued. I believe the greater part of this audience could define this commodity in a manner at least satisfying to themselves. The word "cement" is sufficiently indicative and well enough understood to enable practically all to assert it to be a bonding material. But "portland" fails entirely to be a leading word and enlightening enough to permit one to make an intelligent guess. Indeed, this latter word has historical significance alone and was used originally to identify a new type of cement on the basis that its hydrated products had a fancied resemblance to Portland limestone in England. If you held that this cement is an inorganic one and thus distinguish it from the organic cements or glues or adhesives, you would have placed it in its proper and more restricted class. But we would be far from knowing what it is.

The standards of this Society, moreover, do not help much in this matter. Until 1938 all its cement standards contained a paragraph indented as "definition." The assembly of words contained therein indicated in a very general way how this commodity should be produced. Nothing was stated regarding how it should function in use. Only by assuming that the defined material was to meet the requirements later stated in the specifications could one form a very general idea as to how it is supposed to act. Since that date the definition as such has been dropped. In reality one cannot define portland cement in the manner used by writers of dictionaries. The possible best that can be done is to refer to it as largely a mixture of finely ground synthetic highly basic lime silicates with which are present lesser amounts of highly basic lime aluminates, lime alumino-ferrates, and solid solutions of these with (or compounds of) the many adventitious oxides found in clays, limestones, and the other raw materials used in cement manufacture. The chemical analysis of this heterogeneous powder must show it to be within certain specified chemical limits. It must also be capable of reacting with water to form a bonding agent able to meet certain specified physical tests. Needless to say this "possible best" definition is quite inadequate.

The History of Portland Cement:

It is about as difficult to give the history of portland cement briefly and correctly as it is to define it. Although a patent was given to Aspdin in England, in 1824, to make a product which would now be considered portland cement had he actually made it, the compositional nature of this material was well known before then. Aspdin really happened to be sufficiently commercially minded to patent what others, unknown to him, had already indicated would be a valuable hydraulic bonding material. Vicat in France, and John in Germany, ten to twelve years previously had announced facts similar to those upon which Aspdin based his patent claims. Further, Vicat called attention to the hard clinker which remained after burning and hydrating the impure limes used in making hydraulic lime. He also pointed out that this hard clinker, which was within the range of compositions of our present high-limed portland cements, would on grinding set and harden in a way superior to the cements then available. But he added that he doubted if this superiority was sufficient to justify the extra grinding costs and let the matter drop at this. It should be remembered in regard to Aspdin and his product that his patent did *not* call for heating to "incipient fusion"—the positive criterion of portland cement which so long graced this Society's standards for this commodity. His patent called for decarbonation, since it stated "calcine . . . till the carbonic acid is expelled." Hence, he produced but a synthetic hydraulic lime, which differed from the hydraulic limes then in use in that it was reduced to a powder by grinding rather than by partial hydration.

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[1] Read on June 26, 1940, before the Annual Meeting of the American Society for Testing Materials, Atlantic City, N. J.

[2] Chief, Clay and Silicate Products Division, National Bureau of Standards, Washington, D. C.

Since it appears very probable that the heating of the raw materials to a true clinkering was not put into effect until ten to twenty years after Aspdin obtained his patent, we can place an age of about 100 years on portland cement. Do not conclude that hydraulic cements (those which harden under water) were not known earlier. Early Roman structures show the use of such products. Those used then were mixtures of lime and volcanic ash, later mixtures of lime and calcined ground clay. At some unknown period someone burned limestone containing silica and alumina as impurities and found he had what we would now class as a hydraulic cement and might be referred to either as hydraulic lime, natural cement, or Roman cement, depending upon manufacturing procedures.

In the United States the first hydraulic cements produced were of the natural type and originated in New York State in what is referred to as the Rosendale District. Later the industry spread to the Lehigh Valley of Pennsylvania. In both of these sections an impure limestone was available which, with no additions before burning, yielded on grinding after burning an excellent hydraulic cement. With the introduction into the industry of the rotary kiln, a distinctly American development which has had world-wide adoption, we have the real start of the portland cement industry in the United States. This development necessitated the grinding of the raw materials before introduction into the kiln. This readily permitted of maintaining uniform composition of the raw material through analyses and adjusting and the blending of two or more raw materials of somewhat different composition. This blending and grinding of raw materials is the outstanding distinction between natural and portland cement.

It should be of more than passing interest to note that Brooklyn Bridge stands upon foundations of natural cement. As the true natural cements in this country were gradually but never entirely displaced by true portland cements, there was a period of blending the two. A noteworthy use of such a blend is the elevated road approaching Broad Street Station in Philadelphia.

What Is Portland Cement?

Since this brief historical background in no way indicates what portland cement is basically, it becomes needful to give some time to presenting certain of the known facts and many of the uncertainties in this respect. I have referred to hard lumps of clinker noted by Vicat in his production of hydraulic lime. About 60 years later a young Frenchman spent his early life near a cement plant in France where such hard lumps were still being obtained but now ground to a fine powder and sold as grappier cement. He became so interested in these that he chose a study of them as the basis for his Doctorate degree. As a result we have the early classic piece of research on the nature of portland cement entitled, "Experimental Researches on the Constitution of Hydraulic Mortars," by Henri Le Chatelier who later became such an eminent teacher and authority in pyrometry, metallurgy, and many fields of industrial chemistry, and who remained until his death a consultant to France's largest cement producer.

Although many other earlier and later workers have made valuable contributions on the nature of cement, they will be passed over. Le Chatelier's work is so conspicuous in that with what would now be considered rather inadequate observations and data, he indicated that the major constituents of cement were those that much refined exacting work has since confirmed. He prepared tricalcium silicate ($3CaO \cdot SiO_2$) and believed it to be the only essential constituent that contributed to the hardening of cement. He also prepared dicalcium silicate ($2CaO \cdot SiO_2$), tricalcium aluminate ($3CaO \cdot Al_2O_3$) and other lime aluminates, and what he believed to be a tricalcium alumino-ferrate ($3CaO \cdot Al_2O_3 \cdot Fe_2O_3$) compound. He also called attention to the colored flux which fills the voids between the crystals in the clinker. Hence, he was aware of the four major constituents ($3CaO \cdot SiO_2$: $2CaO \cdot SiO_2$: $3CaO \cdot Al_2O_3$ and $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$—if we assume his $3CaO \cdot Al_2O_3 \cdot Fe_2O_3$ was what is now known to contain more lime) now for some time accepted as being the major compounds present in portland cement, as well as the flux or glass which so many still hesitate to acknowledge as a prominent part of cement. He believed that only the tricalcium silicate played any significant part in the hardening processes, and that "the dicalcium silicate can only take a secondary part, if any, in the hardening of cements." Also in spite of his leaving the tricalcium aluminate mostly out of the picture as a clinker mineral, he goes into some detail regarding its hydration and its effect on disintegration of cement under unfavorable conditions.

The most excellent work done abroad and in this country since Le Chatelier's time has confirmed most of his findings and extended them to give the proper place to the dicalcium silicate and the lime alumino-ferrate, but has left the tricalcium aluminate and the flux (or better glass) still conspicuously uncertain. Uncertainty still remains as to the nature of the role of the always present so-called minor oxides. These include MgO, Na_2O, K_2O, FeO, TiO_2, Mn_2O_3, and P_2O_5 which will average a total of about 4 per cent for the cements of the entire country, but in some cases will amount to 6 per cent. They should be considered minor only because our knowledge of the nature of their existence is meager or minor. They constitute a total that should justify our being concerned about accepting the premise that they may be ignored (especially in studying the physical properties of cement); that the only compounds in cement are the two lime silicates, the lime aluminate, and the lime aluminoferrate; and that the latter compounds exist in a pure form, calculable as to amounts from the percentage of their component oxides found by analysis of the cement.

In the past several decades investigators have studied such ternary systems as $SiO_2—Al_2O_3—CaO$: $CaO—MgO—SiO_2$: $CaO—FeO—SiO_2$, etc., parts of the quaternary system $CaO—SiO_2—Al_2O_3—Fe_2O_3$ and other oxide systems of interest to those studying cement. Data concerning the phase equilibria in these have been painstakingly obtained by not only one worker but frequently by several. The compounds noted were critically examined and their chemical, microscopical, and X-ray characteristics determined in accurate detail. By means of such data it may be possible to predict the state of equilibrium in clinker, *provided* that among other criteria the conditions of heating and cooling and the maximum temperature of burning are known and on the premise that all the lesser oxides in the mix have no effect upon the equilibria of the four major ones. Those of you who are metallurgists and know the tremendous effect on equilibria of slight changes in temperature and rates of cooling and of minor constituents of the melts will readily appreciate the questionable accuracy of predictions not carefully considering such effects.

It is much to be regretted that the procedure adopted by Törnebohm in the latter part of the last century and for quite a period followed by others abroad was not adopted by students of cement, particularly in France and the United States. He, instead of applying chemical formula and names to the crystals he noted in sections of clinker, followed the practice of mineralogists and described them under such names as alite, belite, celite, etc. Although the mineralogist may assign a chemical formula to a mineral, neither he nor others familiar with mineralogy assume that the mineral is of the purity indicated by the formula. He does not assume that an excellent crystal identified as the feldspar orthoclase to which the formula K Al Si_3O_8 is assigned contains these elements and these only in the amounts calculable from the formula. On the contrary, he knows that adventitious elements are present. The mineralogist assigns the formula Al_2O_3 to each of the precious gems ruby, sapphire and emerald, and he does not need to recall that marked difference in physical property—their color—to remind him that they are not chemically pure Al_2O_3. Hence, due to the unfortunate idea of so many that the compounds seen in, or reported to be in, or calculated to be in commercial portland cement clinker must be of the unquestioned chemical purity indicated by their formulas, I am compelled heartily to support the plea of Forsen, made at the Symposium on "The Chemistry of Cements," held at Stockholm, 1938. He states:

> "I also wish to take the opportunity to touch upon a question of nomenclature. The clinker minerals are formed in a similar way to many natural ones, and we know that these generally are contaminated with impurities. We also know that alite differs somewhat from pure $3CaO \cdot SiO_2$ with regard to X-ray pattern—maybe on account of a certain content of aluminate, that belite can be yellowish, possibly on account of a certain iron content—and that celite probably contains some MgO. . . . It appears to me that the existing differences between the pure clinker components and the minerals present in clinker—especially between $3CaO \cdot SiO_2$ and alite—argue in favor of retaining the old names alite, belite, and celite for the clinker minerals and reserving the chemical formulae for the synthetic compounds. . . ."

Reactions of Cement with Water:

It is not the intention here to go into a discussion of the chemical processes of hydration.[3] The investigations in this field have been many and the conclusions figuratively far outnumber the investigations. But from the mass of theories propounded it is possible to pick out a consensus regarding the resultant of the hydration phenomena as manifested by the rates of setting, hardening, evolution of heat, of attack by aggressive salt solutions, etc. Please realize that in the previous sentence the phrase "rates of setting, hardening, evolution of heat, . . ., etc.," has a definite significance. When once portland cement is mixed with water, it were better not to speak of hydrated cement, but always rather of hydrating cement. The reaction is one which continues, if water can have access to all the grains, until the constituents are broken down into the hydrates of the oxides originally present (or carbonates of these if in some cases carbon dioxide is available). Then cementitious products no longer remain. Hydration extends from a minimum of no commercial significance through an optimum of greatest value to a maximum—again of no commercial value. Hence, all the physical properties must be defined as of a specific stated time.

The studies of the rates of the reactions of water with the impure compounds referred to as being present in ground clinker enable one to assign approximately quantitative relative values to each of these for each of the physical properties. Thus, the rate of hydration of the lime aluminate to which the formula $3CaO \cdot Al_2O_3$ is assigned is more rapid than any of the other compounds now identified in portland cement. (Parenthetically, we are stating this fact in this manner because judging by the extremely rapid rate at which the alkalies Na_2O and K_2O can be removed from cement, it would seem that the compounds, as yet unidentified, which contain these hydrate more rapidly than the just cited aluminate.) Next in order is the lime alumino-ferrate (to which the formula $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ is given), then the high-limed silicate (more or less impure $3CaO \cdot SiO_2$), and lastly the low-limed silicate (possibly even more impure $2CaO \cdot SiO_2$). There is left out of the discussion almost entirely the role of the "glass" even though this may exceed in amount that of both the aluminate and alumino-ferrate. This is necessary because of our dearth of information regarding the nature of the hydration of this, at times the third in amount, constituent of portland cement.

It is desirable to consider next the physical properties attributed to the hydrated products of each of the cement compounds. Much of our information in this respect has been obtained from the pure compounds prepared from pure oxides. We can formulate from such data a rather good idea of the relative part each compound may contribute to any one physical property in a commercial cement, neglecting the "glass," which most unfortunately must be left out of the present picture. The high-limed aluminate, which at times is very difficult to detect in clinker either microscopically or by X-rays and in the majority of cases impossible to detect by these critical means, reacts so rapidly that all its physical effects are attained in about 24 hr. In the pure condition it reacts as rapidly as ground quicklime. Its total heat of hydration is approximately 200 cal. per g. and practically all of this is evolved in 24 hr. The pure lime alumino-ferrate evolves about half this quantity of heat, and requires approximately a month to do so. The high-limed silicate in about one month evolves nearly all of its total heat of 120 cal. per g., while the dicalcium silicate has generated but little at the end of 7 days and requires a year to develop all of its total heat of approximately 60 cal.

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[3] Herein hydration is used as including hydrolysis.

From such data it is possible to estimate very approximately the heat of hydration of any cement from its chemical analysis. If the cement is high in alumina and lime and low in silica and iron oxide, it will contain predominantly the high-limed aluminate and silicates and hence will generate a large amount of heat. In large masses of concrete where the interior conditions approach adiabatic ones, a high temperature will be reached at about the same time rigidity will be attained. As the exterior cools to atmospheric temperatures the resulting differences in volume between the exterior and the interior may cause cracking. Hence, if one wishes to remedy this condition one of the ways which suggests itself is through such chemical limitations as will bring about the presence of those constituents in the cement as have low heat of hydration and rates of heat evolution. However, to be sure that this result has been obtained the heats of hydration at certain specified ages should be determined by calorimetric methods.

Although recently the heat of hydration of cement has been discussed so much due to the great amount of construction of dams where the sections are massive, the strength which a cement may attain on hardening still occupies our attention to the major degree. The pure cement compounds have now been studied for several decades as to the rate of attaining strength. Also a large number of carefully chemically analyzed commercial cements have been made into specimens whose strengths have been determined at many ages. Also such other properties as volume changes, resistance to freezing and thawing, heat of hydration, etc., have been ascertained. The percentages of the four major constituents in each of these cements were calculated by methods based upon a number of premises, outstanding among which are that the major oxides combine to form pure compounds, and that the minor oxides do not combine with the major ones or enter into (or influence the properties of) the major compounds. Then equations have been set up for each cement in which the sum of the percentages of each constituent equals the strength, heat of hydration, volume change, or what have you, at any age. Having at hand as many equations as cements, the contribution of each compound to any property has been calculated by the method of least squares.

As a result of the use of both of the above procedures, there are at hand many data of use in predicting the physical characteristics of a hydrated cement from a chemical analysis of the non-hydrated material. From such data we know that the lime aluminate or the lime alumino-ferrate develops so little strength that the alumina and iron oxide may be ignored in this respect. The high-lime silicate, and consequently a high-lime low-silica cement, attains the greater part of its strength in a month; while the low-limed silicate unless extremely finely ground and in the presence of other materials which act somewhat catalytically, at the same age has developed hardly a tenth of the strength of the other silicate. By the end of a year the strength of the two types of silicates shows practically no difference.

We will continue further in this phase of our subject, which to many is so trite, only with some remarks regarding durability, both in reference to natural weathering agencies and to aggressive salts, such as soluble neutral or acid sulfates. Here our data are much more meager than in the case of strength or heat of hydration. But they do show such a trend that we can conclude that many of the commercial cements would be improved as to durability if the alumina and iron oxide were reduced to a minimum that in some cases would cause manufacturing difficulties. The relative value of the two silicates in respect to durability is known with less assurance. Possibly if all concrete, regardless of whether made of a high- or low-silica cement, could be protected for six months and ideally cured, it would be found that thereafter the two silicates would deport themselves alike in regard to permanency.

New Specifications for Cement:

Those of you who have been reasoning a little faster than our subject has been developed have already concluded that in so far as judging the quality of a cement or in so far as the writing of a specification for this commodity is concerned, all that is needed is the use of chemical analyses and chemical limitations. It is to be confessed that there are a goodly number who apparently believe just so. If we will critically and unbiasedly examine the wealth of data at hand, we will conclude that such is not entirely the case and also that there is not only an outstanding lack of the needed physical tests but also an equally outstanding lack of effort to develop such tests. This is due to the fact that within the past decade there has been brought to the fore, and recently to a head, the agitation against one standard for cement and for the recognition of standards for five types. It happens that within that period, having started with emphasis on chemical composition, the discussion has centered around that thought. The need of physical tests was recognized, but thought of the difficulties to be encountered in developing them at all times prevented overcoming the inertia of getting under way. But now at this annual meeting, with the Society considering the acceptance of tentative specifications for five types of cement in which chemical requirements are so outstanding and physical requirements so dimly visible, it seems time to start active work in developing physical tests despite the visioned difficulties—and the difficulties are more of vision than of fact.

As Chairman of Committee C-1 on Cement, I have called the committee's attention to the fact that the new tentative specifications are a striking outline of the needed active work. Without speaking disparagingly of the new specifications, may I say that they are as good as an outline of research, through their inadequacies, as they are good purchase specifications,—and they are good purchase specifications regardless of their inadequacies. But more of the standards later, and before considering them, may I be pardoned for presenting one more thought regarding chemical composition (or compounds), physical properties and specifications in general.

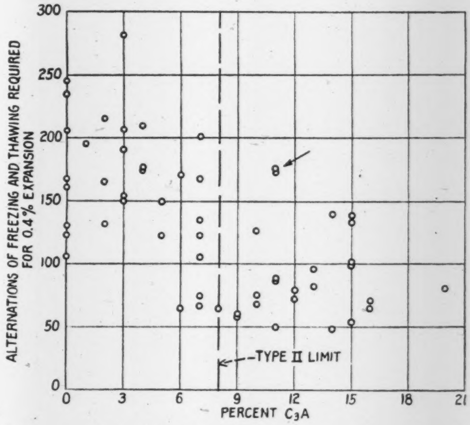

FIG. 1.—Relation Between C_3A in Cement and Resistance to Freezing and Thawing.

For the purpose of illustrating my

thought, there is presented a graph (Fig. 1) through the courtesy of the Public Roads Administration, which it prepared from some data obtained from the laboratory of the Portland Cement Association. In this graph the relation between the calculated lime aluminate content of some cements and the expansion of some specimens of these subjected to alternations of freezing and thawing is shown. This was used in part in aiding in setting the limit on the permissible amounts of the calculated $3CaO \cdot Al_2O_3$ in the proposed tentative specifications for type II cement. These data have been used to show:

1. There is a positive trend in the relation between the amount of this compound and the resistance to freezing and thawing,
2. There is a positive lack of trend in this relation,
3. There is a trend sufficient to justify the use of the 8 per cent limit presented in the new standard.

Now, let us lay aside our role of a specification writer guarding the interests of the consumer and put ourselves in the position of the manufacturers of the two cements indicated by an arrow. These are shown to have approximately 11 per cent aluminate and to have withstood 175 cycles of freezing and thawing. Certainly we can rightly protest the 8 cent limit since, although our products are 33 per cent in excess of the permissible limit, they are better in the freezing-and-thawing tests than the majority of the cements within that limit. Then back in our role of specification writer we would tell the two producers that granted that their contention is correct, the large majority of the cements exceeding the limit showed a much more unfavorable deportment in the physical test than those within the limit. Further, we can inform the two producers that we cannot adopt limits which will permit a few excellent exceptional cements to be accepted when by so doing we allow the use of a much larger number of proven inferior ones. But in a third role—that of an investigator and a curious one in addition—we look at the data and say, without hesitancy or questioning, that something is wrong,--if we hold (and we must) that the only variable in the specimens was the aluminate content of the cements and that all specimens were similarly tested. Back in the role of producer we would demand that we be shown without question that our cement contained 11 per cent aluminate. We would not accept calculated values. Finally, as a specification writer, we would have to confess that we have no way of meeting such demands and hardly half a dozen workers in the country are actively engaged in trying to solve the problem.

One can go through the many publications on cement and concrete and find much data and many graphs similar to the one presented. There will invariably be found the outstanding exception or exceptions. These latter will receive but scant attention regardless of the fact that they may approach the ideal far more closely than does the average or those accepted as indicating the trend. It is believed the majority of you—and especially those not familiar with the cement or concrete field—will agree that the data are at fault. Not only is it likely that the exception is not an exception, but also it is quite probable that the average is not the average. In the case cited it is extremely likely that there were other variables in the cement, in spite of the accepted premises, than the aluminate and that we are not justified in drawing the conclusions we do from data such as these, which are far from critical.

The new tentative specifications, which it is hoped the Society will accept at this annual meeting, represent quite a departure from those which remain in the Society's volume of adopted standards. These latter cover a "Standard Portland Cement" and a "High-Early-Strength Portland Cement." The tentative standard even though it will appear when accepted under a single serial designation will be a standard covering five types of cement. Type I is practically the same as that covered by A.S.T.M. Specifications C 9 – 38; type II is quite similar to Federal Specification SS-C-206, wherein it is referred to as a Moderate Heat of Hardening Cement; type III, a high-early-strength cement, is essentially the same as A.S.T.M. Specifications C 74 – 38; type IV is quite similar to recent specifications of the Bureau of Reclamation for the purchase of Low Heat of Hardening cements; and type V is very closely Federal Specification SS-C-211, wherein it is referred to as a Sulfate Resisting cement.

The question immediately arises—are such types needed and can you produce from commonly and cheaply available raw materials such ranges in SiO_2, Al_2O_3, Fe_2O_3, and CaO (and the minor adventitious oxides) content as will yield these different cements? The answer to this question, according to general consensus, is yes. But let us go a little further into this matter.

Portland cement in this country was a matter of but minor commercial import prior to the beginning of this century. Yet by 1908, the U. S. Geological Survey had a laboratory at Atlantic City studying the effect of sea water on various cements and concretes. This laboratory had been established through the cooperation of what is now the Portland Cement Association because there was already much questioning of the durability in sea water of the portland cement then on the market. Included among the cements studied were four of an especially high iron oxide content, three of which were of domestic origin. The fourth had been made in Germany under patents held by the Krupp interests. One of the claims of the patents was that cements of high iron content resisted the attack of sea and other sulfate waters to a greater degree than those of the low Fe_2O_3 and average Al_2O_3 content then mostly produced. In the 1908 *Transactions* of the American Society of Civil Engineers[4] there appears a paper and a discussion of the evolution of heat by the cement in concrete dams and its significance. It is hence quite evident that at this very early period (32 yr. ago) in the history of portland cement in this country the inadequacy of the one and only type of cement available in its relation to sulfate resistance and evolution of heat was a matter of much critical interest, and that the need of other types was not questioned by many—including some producers.

The question of the availability of raw materials and the possibility of commercially securing the ranges in composition that will yield cements meeting one or more of the five sets of requirements demand some attention. It is granted that in order to make all five types most manufacturers will have to bring to their plants varying amounts of raw material now not at hand. With the needed materials at hand it is possible to blend them so as to secure in the finished product the composition required by the specifications. But this does not necessarily mean that at all times these compositions will yield the values demanded by the few physical tests. Neither does it mean that other compositions will not at times give the required physical results and hoped-for job results. It does, however, mean that at the present stage of the development of the knowledge and the art of making cement we have not either completely correlated composition and physical properties or completely correlated chemical composition and all the compounds actually present in the cement. However, in the present stage, the specifications must give attention to and base their requirements, within limits, on the deportment of the majority rather than on the exceptional minority, —even though it is granted that some of the exceptional minority in certain respects may be the more desirable. This applies particularly to the general cases of usage. In certain special cases, the choice of the ideal exception should apply.

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[4] Charles S. Gowen, "The Effect of Temperature Changes on Masonry," *Transactions*, Am. Soc. Civil Engrs., Vol. LXI, p. 399 (1908).

Granted that at times there is a lack of relation between composition and physical properties, one can be assured that the range in compositions made possible by the proper blending of raw materials will yield cements quite different in physical properties. One of these cements when properly chosen for a special service can be used with good assurance that better results will be obtained, if properly made into mortars and concretes, than by the use of one cement for all services. The user, therefore, has at hand five different products from which by intelligent use he can accomplish more satisfactory results than before. He must determine which type he should use in any case by studying the conditions under which he will use the cement, the conditions during its later life and the requirements in the standard wherein the nature of the several types is given. In many cases the choice is simple and can be quickly made. A high-early-strength cement would hardly be selected if there is no need for haste in construction or in placing the finished structure in use. A low-heat or a sulfate resisting cement would hardly be selected for a reinforced concrete frame building to be covered with brick or stone. There might be some question as to the choice of types I or II. In an increasing number of cases there is now no need for hesitancy, as the type I made by a considerable number of producers is identical to their type II, and this number will increase with time. But if experience has shown anyone that in general work he has obtained excellent results through the use of a cement which is truly type I, he had better continue with it rather than try a type II product. Types IV and V are for use when low heat of hydration and sulfate resistance, respectively, are of importance. They are special types which will not generally be kept in stock by producers, at least for some time in the future.

A word of caution must be given regarding the choice of cements for special uses, such for instance, as resistance to sea water. Can it be assumed if a type V cement is used for sea water construction that *without question* our hopes for a very extended successful service will be realized? The answer is no, but even with such an answer type V should be used. A positive yes cannot be given to such a question. Concrete is not composed of cement alone and it is the other familiar components—and workmanship in particular—which in most cases are the causes of trouble and which compel the negative answer. But one can be certain that in the unfortunate cases where poor concrete may be made, the use of a cement of type V will very likely be an assurance of a greater life than if type I, II, or III had been employed.

Degree of Grinding (Fineness):

The proposed new tentative specifications bring back into the standards of our Society an old friend who disappeared for a few years—the requirement covering fineness of grinding of the finished product. While this requirement might be welcomed back as an old friend, it surely caused in times past much unfriendly discussion. Whether there should be limits for fineness is a much mooted question. Requirements of this kind surely are within the class of those which tell the manufacturer how to make his product and as such are among the least acceptable of specification details. It would seem that within reasonable limitations the manufacturer should be allowed to grind to whatever fineness yields the specified physical properties that are affected by the degree of comminution. The physical properties mostly affected by fineness are time of set and strength. Thus, a low-heat cement can be brought into the class of type I cement by grinding alone. The only distinction between many high-early-strength cements and type I cement is that the producer has taken his more "choicy" type I clinker and ground it about 1000 sq. cm. per gram finer than he would if making type I cement.

It is further known that it is not necessary to grind all kinds of clinker to the same degree of fineness to obtain the same strength. Why, therefore, not allow the producer to use whatever fineness will yield the strength indicated for each type? Why compel all to grind to the same minimum fineness? The candid answer is that many believe that fineness is closely associated with other properties, mostly undesirable, which through our ignorance we cannot cover by test methods or limitations. For instance, many believe that fineness is intimately related to volume changes during hardening. Too finely ground cements are believed to require undue amounts of water and consequently undue shrinkage and cracking may result. But to meet such a viewpoint there should be a maximum limit and not the minimum limit which appears in the specification. Others feel that the coarse particles of the less finely ground material may hydrate later and cause disruptive expansion. Some question whether the very fine particles do not hydrate so promptly during mixing and placing that they confer nothing to the strength and may tend to be concentrated in any laitance formed or on surfaces against forms where they rather promptly cause map cracking or crazing, or unsightly weathering. However, as no one has brought forward suitable test procedures and specification requirements, the obviously unfortunate procedure has been followed of "saying it with fineness" and believing that by so doing we have not lessened the chances of getting a better cement but possibly increased them.

The Autoclave Test:

It is difficult to set a date when cement or cement products were first submitted to elevated temperatures and pressures within a few hours or a few days after mixing with water. There are possibly some very few in this audience who will remember a meeting of the Society about 30 yr. ago, when a paper was read in which the conclusion was drawn that a better cement would be secured if neat specimens after 24 hr. in a saturated atmosphere were submitted to a steam pressure of 295 psi. and would show not more than a certain volume change or reduction in tensile strength. A very lively discussion resulted which did not quiet for several years. Some few cement producers have continued to use the device in following their plant production. Some cement products have always been cured by steam at atmospheric or higher pressures. Also a number of investigators have studied the effect of the high-pressure steam on the nature of the hydrated products formed under such conditions and how this treatment has affected the durability of these products when in contact with solutions of sulfate salts.

About three years ago considerable surprise was occasioned by the proposal that there be introduced into the Society's standard specifications for portland cement, a maximum permissible expansion of 1.0 per cent as a result of submitting a neat cement bar, 24 hr. old, to a steam pressure of 295 psi. for 5 hr. This proposal originated with the producing interests who felt such an accelerated test would eliminate cements which would otherwise satisfactorily meet the standards, but which would show in service too great an expansion at a period of from 5 to 10 yr. after fabrication. Again, a very lively discussion arose and last year at a session of the Society negative action was taken. In the proposed new tentative specifications it will be noted that an autoclave test is proposed with a maximum limit of 0.5 per cent for all types of cement.

Whether or not a test of this nature should be in our standards and what the significance is of the test is still a debatable matter. You can rest assured, however, that the requirements cannot in any manner jeopardize the consumer's interest. Even under the high limits for the expansion specified he is given additional protection so far as quality is concerned. Few producers have any difficulty in making "autoclave cement" when using the compositions required by types II, IV, and V. It may interest you to know that the low-heat cement being purchased by the Bureau of Reclamation must not exceed a maximum expansion of 0.2 per cent according to this test. This is 60 per cent less than that allowed by the Society's proposed new tentative specifications. Some producers did have difficulties in meeting the demands when making cements of types I and III, but they have learned that, among other things, more rapid cooling of the clinker will result in a marked decrease of expansion in the high-pressure testing in the autoclave. Incidentally, this is an interesting illustration of certain phenomena not discussed in this talk, namely, changes in the character or structure of the clinker induced by manufacturing processes which cause changes in the physical properties of the cement. These structural changes in the clinker are not accompanied by calculated compositional changes.

The autoclave should be a matter of moment to the producer—and it has been such. It taught a number very promptly that closer control at certain stages in the course of manufacture would materially reduce autoclave expansion. From all viewpoints this test must be credited with having introduced some desirable technique and control into the production of cement and must be acknowledged as yielding additional assurance of quality to the users.

In much of the discussions so far regarding this test there has been introduced too much of a positiveness concerning the cause of the expansion induced and the significance of the test. This has tended very strikingly to deflect attention from the test itself. It is better at this time, and in the future until basic research has yielded much of a positive nature, to confess that adequate data are not at hand to indicate the nature or the difference in the nature of the cement-water reactions at normal and elevated temperatures (and pressures), especially as to how *all* the constituents of the cement are affected. Therefore, no positive answer can be given as to why certain cements expand more than others when treated in the autoclave. Whether or not a high autoclave expansion without question signifies a large expansion in later life is also still to be proven. That the test assures generally a more uniformly and better made cement is without question.

Resistance to Weathering and Sulfate Action:

Among those interested in highway construction, much thought has been given during the past decade to the resistance of concrete to the natural weathering agencies. To the effect of the natural agencies there must be added at times others, such as the use of calcium chloride on ice-covered highways during the winter season. Regardless of the extended work that has been carried out in the hope of elucidating the relation between chemical composition and service under such conditions and in spite of the efforts to develop adequate predictive tests, no acceptable physical test suitable for such use in specifications has been brought forward. It is therefore necessary to continue to place our reliance on the results of the chemical analyses, the strength of the small specimens and the newly added autoclave test. Yet it is known that at times these have not resulted in our securing the hoped-for good quality concrete—even when we allow for all the other defects which may get into concrete, such as possibly bad aggregate, improper amounts of water, and inadequate workmanship of one kind or another.

Thoughts very similar to those just expressed apply regarding the resistance of cements to aggressive solutions of salts. Although the effect of such agents on cements and concrete have been the subject of far more publications than the effects of weathering agencies, the solution to the problem and adequate test methods do not appear to be any closer at hand. If we would accept without question the most frequently followed premise, namely, that the aluminate of lime forms complex sulfate salts having a much greater volume than the aluminate compound in the cement, then there should be a possibility of settling the issue by limiting the alumina or aluminate content. But even though this procedure is followed in the specifications to a certain extent, we are immediately faced with the fact that the true answer is not so simple. Many structures of high alumina and aluminate cements exist in unfavorable sulfate surroundings and have met all hopes. The Statue of Liberty in New York Harbor stands on a foundation of a quite high alumina cement. The existence of such successful structures controverting our hypotheses unquestionably compels us to doubt their general validity. Indeed, we are compelled to acknowledge that in many cases we must look elsewhere than to the aluminate for an explanation of the vulnerability of concrete to sulfate solution attack. However, we are again compelled to follow the usual procedure, in lieu of anything better in the nature of adequate test methods, of penalizing the few cements of acknowledged good field record but which do not fit into our at times faulty hypotheses and specification requirements that low-alumina and low-lime-aluminate cements give better results in contact with sulfate waters than cements of high aluminate content. Once again we become painfully aware of the marked need of research and of the development of test methods.

Admixtures:

As a red flag is purported to be to a bull, so were admixtures to some cement producers. Please note in the foregoing that "admixtures" is used as in the past and "purported" is dropped in the last part of the sentence. Until very recently anyone who would dare propose the use of admixtures with portland cement to improve any somewhat lacking property was immediately figuratively killed with anathemas hurled by some producing interests. Then almost overnight (and a relatively recent night) there was a complete reversal of attitude on the producers' part. Now many of the latter are actively studying the effects of various kinds of admixtures as reflected in increased durability and quite a number are placing on the market cements containing such additions.

There never have been any logical reasons for this peculiar adverse attitude. The fact that all cements contain 3 or more per cent of gypsum, added during the grinding of the clinker as a necessity to give a product meeting the time-of-setting requirements, has never made producers open-minded towards the possibility of using other admixtures to enhance other properties. The first breakdown of this attitude came a few years ago as the result of the marked ability of an organization to demonstrate that one of their products added to the clinker before grinding very materially reduced the cost of grinding. As a result the Society's standards, first for high-early-strength cement and in 1938 for portland cement were amended so that under certain conditions admixtures are permitted. Bear in mind that before this action a portland cement according to the Society's standard was only such if it contained no additions of any kind or amount other than calcined or uncalcined gypsum or water.

There is now agitation to permit the use of other admixtures. Some of these have been found most advantageous according to recent field and laboratory tests in increasing the life of concrete in highways subjected to "sanding" during freezing conditions with calcium chloride. Most of these are of such a nature that frothing takes place during mixing and consequently as much as 10 per cent or more of air may be introduced into the wet concrete. This gives a readily placeable mix, which before setting "bleeds" or releases water to a negligible degree. Like the grinding-aid admixture first cited, these latter are used in very small quantities—mostly less than 0.05 per cent of the weight of the cement.

It is to be hoped that, with these two obvious examples before us of the betterment of cement through the use of admixtures, all concerned will have a more open mind in this matter. There is no reason to assume that improvement in other physical properties cannot be secured by means of other additions. The setting and hardening of cement is a chemical reaction and in such all recognize and have before them many examples of the marked alterations in reactions induced by the presence of small quantities of foreign materials. It is granted that at times such reactions may be adversely affected by the small amounts of additions—as in the particular case of cement, the marked slowing of setting and hardening induced by very small quantities of solutions of sucrose or lead or zinc salts. But such positive results even though adverse should only seem to confirm the thought that there must be other materials which can cause most advantageous effects.

Again, let us bear in mind that the setting and hardening of cement is a chemical reaction and further one which takes place very slowly. If we accept the belief of many, and some of us will not, that cement in the form of concrete is gaining strength continuously no matter what its age, then we must accept the fact that hydration or the reaction of the cement and water is continuing just as long. We know that the rate of the reaction can be materially accelerated by fine grinding of the cement, by heat, and to a less degree by the use of calcium chloride, etc. It does not take much of an imagination to conclude that there are likely to be a host of other materials which will speed up the reaction tremendously, possibly to the extent that within a day we may have the strength usually attained at the end of a year. It is curious that so much time has been spent in developing different kinds of cement and so little on developing very promptly by means of admixtures the full inherent qualities of the cements already at hand.

So far nothing has been said regarding the much mooted colloidal or crystalline nature of the hydration products. Much evidence shows that the bonding hydration products are, like most bonding agents, either organic or inorganic, of a colloidal nature. This does not deny that there are not also formed during hydration some crystalline products. These have, however, no or low bonding qualities. It is known that colloids in many cases may be either water soluble or distended to a tremendous volume in the presence of water. It is also known that these may be so treated that they are unaffected by water. The solubility of egg albumen in water before heating and the insolubility after heating to a certain temperature is a good example of this phenomenon. The colloid formed in the setting of cement has the quality of expanding and contracting as the humidity is raised or lowered to a marked degree. Thorvaldson has called attention to how heat will affect the hydrated cement so that its resistance to sulfate action is much enhanced. Cannot this be looked upon as a kind of fixation of the hydrated colloidal products so that their stability is materially increased? Also can we not believe that there are materials—admixtures if you please—which may so affect the course of the hydration that the resulting products are rendered either irreversible or more stable in respect to other reactive compounds with which they may come into contact during service?

Indeed, the use of admixtures should not be frowned upon or scorned. On the contrary, their development and study should be highly commended and fostered. Accepting cements as they are now produced, there seems to be no more brighter prospect of improving these throughout their entire life—from the time they enter the concrete mixer until progress in the course of time compels the abandoning or demolishing of the structure into which they entered—than through the use of admixtures. The happening upon the effect of a few admixtures of a "froth" producing nature in increasing the life of concrete highways justifies a vigorous search for others possible of enhancing other qualities of cement.

Before leaving this subject may attention be called to a particular direction into which research of this character might be pursued. The excellent qualities of low-heat cements for mass concrete is generally accepted. But such cements have a low rate of hardening and acquiring strength. Indeed, this is so marked that in some cases during cooler weather, in order not to delay work through the need of holding the forms in place longer due to slow hardening, it was necessary to substitute moderate heat of hardening cements for the low-heat ones. Further, if the low-heat cements are also low in both alumina and iron oxide, we have a sulfate-resisting type of cement, which is also slow hardening. Also at this time there is a marked tendency to depart from the high-limed cements to the high-silica ones. These like the low-heat and sulfate-resisting types, unless very finely ground, are also slow hardening. But if this characteristic could be overcome through the use of an admixture acting as an accelerator, we would have approached very closely to the ideal all-purpose cement. There is not much doubt that this accelerator will be found some day—even though it may not for psychological reasons be called an admixture.

Some Proposed Tests for Portland Cement:

Within the past ten years five interesting tests and specification limitations have been proposed for use in accepting cement. These deserve some special comment, but before doing so, it is desirable to say a few words regarding the attitude of those interested in cement towards new tests. It is very proper that newly proposed tests should run a gauntlet of testing and trying themselves. But this should be done in a thoroughly open-minded manner. The new tests should demonstrate that they apply to a characteristic not covered by a test already in use, that they more adequately cover a property than does any used test, or that they determine a property in a simpler way. They should give reproducible results in the hands of the average laboratory operator. They cannot be adopted if they are reproducible only in the hands of the exceptional worker. They should also predict a quality which is of concern in the use of the cement. However, in spite of the recognized lack of such tests for cement, there is too much of an attitude exhibited of damning all newly proposed tests without adequate trial largely because they are new. When half-heartedly tried, there is too much magnifying of all the little molehills of minor anticipated trouble into mountains of impossibilities. This is one reason why new tests are so slowly adopted by those interested in cement.

The late Thaddeus Merriman proposed a test for determining the resistance of cements to sulfate waters. In brief, a specimen in the form of a thin slab of neat cement is suspended in a sodium sulfate solution. This solution which rapidly becomes alkaline is titrated at stated intervals to neutrality with a sulfuric acid solution. The quantity of alkali (including Na_2O, K_2O, and especially CaO) liberated is large at first, then becomes less and more uniform with time until when and if failure is imminent there is a marked increase in the amount liberated. This is accompanied by warping, checking, or cracking of the slab. If this occurs within 28 days, it is taken as indicating poor service. This may be a good acceptance test for selecting a cement to give good resistance to sulfate attack, but unfortunately it has not been subjected to needed coordinated cooperative trials. Although it has been some time since it was first proposed, there is still lacking the tie-in between the laboratory and field trials.

Merriman also proposed what is generally called the "sugar test" for cement. This was developed as a means of ascertaining the degree or completeness of burning of the clinker. Briefly, the test consists of leaching the ground cement with a rather strong solution of sugar (sucrose) and neutralizing the resulting alkalinity. For the cement to be of an acceptable nature the amount of acid required must be below a certain value. Merriman believed that such accepted cements produce a better concrete than that given by other cements not meeting his test and that the concrete contained the maximum amount of what he calls "amorphous constituent"—the desirable hydration product of cement. Although the test was first proposed about ten years ago, we are still lacking the results of an extended series of tests or observations to correlate the findings in the field and in the laboratory. The tests do show striking differences between cements which in other respects are the same. This applies to those from the same mill as well as to those from different mills. It is also generally conceded by producers that cement which will meet this test must be more carefully processed and in some cases quite different mill procedures must be followed than those used for the average cement.

Mr. Ira Paul, of the Division of Engineering of the New York State Department of Public Works, has proposed two tests—a "floc" test and the subjection of clinker fractionated according to certain sieve sizes to an autoclave treatment very similar to that contained in the Society's proposed new tentative specifications.

The remarks made regarding Merriman's sugar test apply to Paul's "floc" test. It is an interesting test which shows marked differences between otherwise similar cements, it has not been correlated with field service and it has not received the open-minded attention that it deserves. Autoclaving of the clinker has much to commend it; indeed, when one gives the proposed test some thought, he is inclined to conclude that the clinker and not the finished set cement should be subjected to the high-pressure steam treatment. This would be particularly true if it can be shown that the hard pieces of clinker are not strained due to the rapid cooling and that the breaking and shattering, which constitutes failure according to the methods, is not a result of the release of the strain in the autoclave. Committee C-1 which has adopted tentatively an autoclave test for cement (ground clinker) should give immediate and intensive attention to the autoclaving of the clinker.

Mr. T. E. Stanton, Materials and Research Engineer of the California State Division of Highways, in attempting to solve the problem of why so much concrete in a certain section of that state gave most inadequate length of life, found that if the concrete was made of cements containing less than 0.5 per cent Na_2O plus K_2O (determined and reported as Na_2O) the results were all that was desired. It has been stated before in this lecture that these alkalies very promptly go into solution in the mixing water. Apparently this alkaline solution acts upon certain minerals in the particular aggregate in a manner that produces such marked swelling that disintegration results. In preparing the new tentative specifications, Committee C-1 gave much thought to Stanton's findings, but felt that at the present time it could not fix limits for the permissible amounts of alkali. You can be assured, however, that much more will be heard about the effect of these oxides, which are present in portland cements in such small quantities (averaging about 0.75 per cent for the cements of the whole country) that but few analyses show their presence, although the analyses will total 100 per cent or more.

The Effect of Plant Processes:

Although so far the discussion has centered largely around composition *versus* physical properties, at times there have been some remarks made which could properly be construed as inferring that there are frequent occasions where there is a want of any relation between composition and properties. Tersely,—it is a fact that occasionally cements of the same oxide composition and calculated compound composition have distinctly different physical properties. All of the reasons for this are not known. But some of the newly proposed tests and some recent research have shown us some of the reasons; others we can only deduce from analogies with other materials and data.

When it became evident that the specifications for low-heat cement for Boulder Dam would be based upon compositional as well as physical (calorimetric) requirements, objection was raised by one producer on the score that by "heat treating" cement of ordinary composition and hence different from the proposed low-heat composition, he could produce a low-heat cement according to the calorimetric requirement. Since then it has frequently been found that by the calorimetric measurements, the heat of hydration differs markedly from that which could be calculated from the composition. Also cements of practically identical composition have far from identical heats of hydration.

With the reopening of the question of autoclave testing of cements a few years ago, some little research soon showed that a rapid cooling of the clinker would in many cases result in a cement which had decidedly less autoclave expansion than the same cement slowly cooled.

Both of these cases bring vividly to us the fact that without making any compositional changes the quality of the ground clinker, evaluated in two widely different manners, has been materially changed through processing subsequent to the attaining of the maximum kiln temperature. To appreciate why this may be the case, it were well to discuss very briefly the nature of the materials reacting in the kiln, what may be taking place therein, and how the state of combination reached at the maximum temperature may or may not be the same as that of the cooled clinker.

Suppose we take as typical the case of using limestone and clay as raw materials. Even though other raw materials might be used, they would differ from these only in the relative amounts of the component oxides. These are ground together and then charged into the rotating kiln. Dehydration first takes place. Then follows, almost simultaneously, decarbonation of the limestone and a breaking down of the clay into free SiO_2, Al_2O_3, etc. Then recombination starts to take place, but mostly through diffusion and to a minor degree through melting and subsequent crystallization. How much formation of glass takes place depends upon very many circumstances—such as the lime-silica ratio, the amounts of Al_2O_3 and Fe_2O_3 and their ratio, the amount of fluxing agents at hand such as MgO, P_2O_5, Na_2O_3, K_2O, etc., the maximum temperature, the length of time at the maximum temperature, etc. Next let us assume that under any one set of conditions the time and temperature of heating have been such that equilibrium has been attained. Under this hypothesis, what is the likelihood of maintaining this state during cooling and corollarially what are the possibilities of altering it? Even a casual reference to the phase equilibria diagrams of the ternary system $CaO—Al_2O_3—SiO_2$ by Rankin and Wright and to its extension into part of the quaternary system $CaO—Al_2O_3—Fe_2O_3—SiO_2$ by Lea indicates that likely the high temperature equilibria has not been maintained and in many cases could not be. Further, one would see that by such "heat treating" as different reheatings and coolings under closely controlled conditions or by different controlled rates of cooling, it is possible to bring about the formation of distinctly different amounts of the compounds.

Examination by the petrographic microscope has confirmed that the different conditions cited give optically different clinker and the ground clinker yields different physical results. It seems quite certain, therefore, that "heat treating" can produce decided changes evidenced by different qualities of the finished cement.

Time will not be taken to refer to other changes in plant procedure which may materially affect the quality of the cement—but do not forget the marked changes, which have been referred to several times, induced by different degrees of fineness. It were well, however, before dropping this phase of the talk to suggest that producers might well look to other types of kilns wherein the burning and cooling might be more carefully controlled. Granted that the rotary kiln has done so much to develop the industry and to make it possible to market a commodity at the customary low price, it seems well to remember that furnace design of all kinds has materially developed since the advent of the rotary kiln. Even the old stack kiln, thrown completely over by the domestic cement producers, could be slightly modified to permit of more uniform operation and control than the rotary kiln. The installation of regenerators or recuperators would reduce it to a very low cost fuel furnace. But the ceramic industry which has so long favored the stack kiln is supplanting it with tunnel or ring kilns, due to their increased fuel economy and ease of exactness of control. Either of these two types would permit of wider ranges of rates of heating and cooling, and the ring type would permit of simultaneous burning of several types of cement requiring different burning temperatures.

Concrete:

Doubtlessly it has been expected that some extended remarks would be made concerning concrete. However, much has been said regarding this applied form of cement and at other times when the word "cement" has been used, concrete could have been used with equal correctness. To go into even minor detail anent this commodity will not be possible now when so much time has already elapsed. But I acknowledge that a few remarks are in order. These I will introduce by quoting a recent statement by Prof. Bart J. Shine of the University of Cincinnati:

> "A wise economist, an elderly one, recently made this statement—Economics is now firmly founded on the doctrine of uncertainty.—I wish that all of the concrete designers in the country could reach that degree of enlightenment because concrete design is not yet firmly founded on the doctrine of uncertainty. What we actually do, I think, may be expressed something like this: We design a structure of an imaginery material which does not and cannot exist. It is an elastic material in which stress is proportional to strain, and it is incapable of taking tension. Then, having determined the dimensions of the members, we erect in the field a structure of a somewhat different material with the expectation and hope, generally fairly well realized, that the ultimate result of our procedure will be a structure which is both safe and economical. But it is not the structure we designed, it does not act as would the structure that we designed, and yet the results are startlingly satisfactory."

Although Professor Shine was referring to the design of concrete structures, his thoughts apply equally well to the concrete to be used in the structure. Would that we more completely realized the uncertainties connected with the production of concrete. We do not seem to realize that, although we have a mass of data (unfortunately too much are of the laboratory type) and have had long and interesting discussions concerning them, the making of concrete is far from an exact science. Take, for instance, the many methods, formulas, procedures, etc., advanced for designing a mix of "x" strength from the cement and aggregate at hand and let us note one feature of their frequent application. It can be seen that in so many of these cases, as long as something better has been obtained than what the theory would predict, it is assumed that therefore there is now no longer any uncertainty regarding what to do to get what it is thought is wanted. Incidentally, having obtained something other and much better than expected no advantage is taken of this better quality. The fact that the application yields an answer of $1.5x$ is cited with great delight as evidencing the positive proof of the validity and usefulness of the theory or procedure which has been followed. (If $0.9x$ had been obtained it is likely that no citation would be made or it would be used as evidence of the lack of validity of the methods used.) Truly concrete mix design is based upon the principle of uncertainty, although it is not acknowledged and it candidly should be.

Can we not also aptly apply Professor Shine's remarks to portland cement as well? Would it not be just as true if he had said in part:

> "A wise economist, an elderly one, recently made this statement—Economics is now firmly founded on the doctrine of uncertainty.—I wish that all of the engineers using cement in the country could reach that degree of enlightenment because cement technology is not yet firmly founded on the doctrine of uncertainty."

Were it so founded many of the statements of the quality of cement and predictions regarding its future deportment in service would not be made with the positiveness and finality which now characterize them. The recognition of the uncertainties in these respects would surely lead to more determined and more effective efforts to secure the results needed to change uncertainty to certainty. In the meantime it must be accepted that cement technology functions upon the bases of uncertainties. But the fact that it is certain that there are still many uncertainties regarding the true nature of portland cement, how it reacts with water and how it will deport itself under certain trying conditions, in no way detracts from the proven findings of its tremendous use that "the results are startlingly satisfactory."

APPENDIX

The accompanying photomicrographs have been selected to enable the reader to visualize the appearance of the several compounds referred to in the lecture as assumed to be present in portland-cement clinker. They enable one also to appreciate some of the difficulties encountered by the microscopist in his studies of the nature of portland cement. In the majority of cases the illustrations were prepared by using light reflected from etched polished surfaces of clinker. In some few cases the photography was carried out by means of light transmitted through polished thin sections.

With a few exceptions the photomicrographs have been prepared and previously used by Herbert Insley, Petrographer at the National Bureau of Standards, in the course of his study of portland cement. Although in a number of instances they appear to be taken of discrete grains distributed upon a plate or cover glass, such is not the case. In all instances relatively large fragments of clinker were sectioned, polished, and very small fields enlarged either by reflected or transmitted light to the magnifications shown in each figure. In the following captions tricalcium silicate is abbreviated to C_3S, dicalcium silicate to C_2S, tricalcium aluminate to C_3A, and tetracalcium ferro-aluminate to C_4AF.

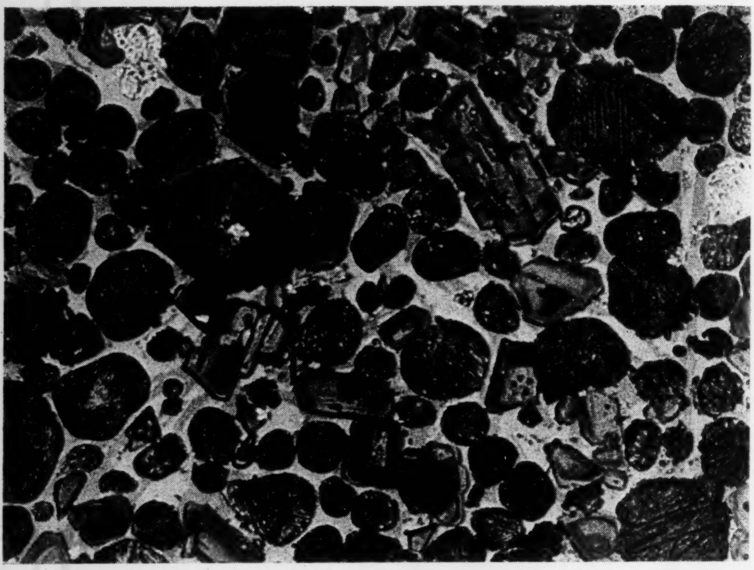

FIG. 2.—A Polished Section of Clinker of a Low-Heat Cement Used in Boulder Dam (× 500)

Etched with water for 2 sec., followed by 1 per cent alcoholic solution of HNO_3 for 2 sec. showing (*a*) angular grains of C_3S with marked zoning (indicating solid solution), (*b*) rounded grains of C_2S with distinct striations caused by twinning, (*c*) light interstitial material (C_4AF), and (*d*) darker interstitial material (probably glass).

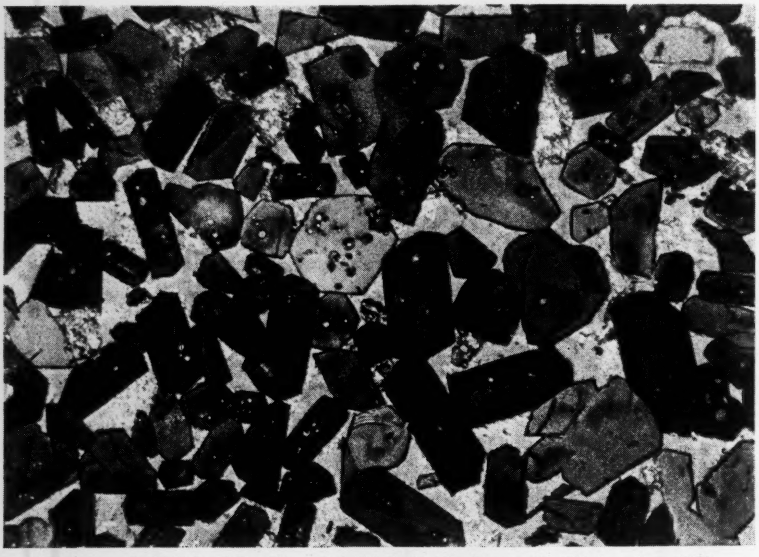

FIG. 3.—A Polished Section of a High-Early-Strength Clinker (× 500).

After etching with water for 2 sec. followed by 5 per cent NH_4Cl for 3 sec., showing abundant C_3S in well-developed crystals with hexagonal outlines.

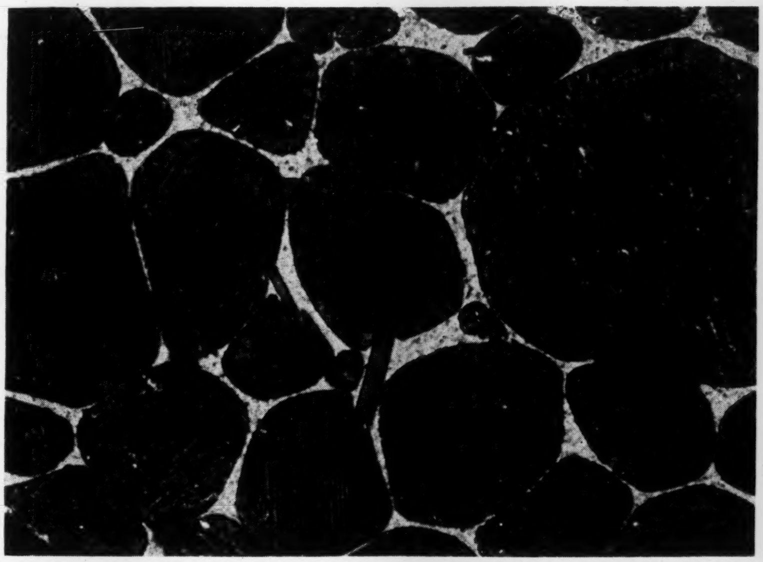

FIG. 4.—A Polished Section of a Low-Heat Clinker (× 1000).
Etched with 1 per cent alcoholic solution of HNO_3 to show the intricately twinned C_2S.

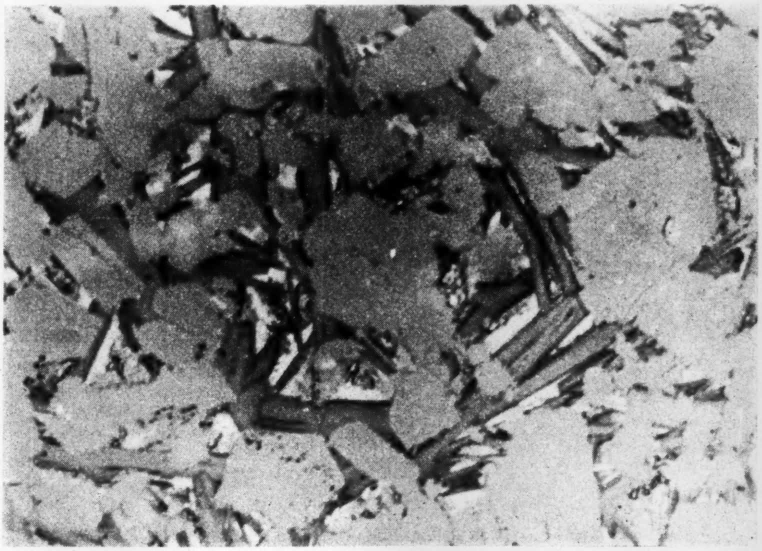

FIG. 5.—A Polished Section of a Normal Clinker (× 500).
Etched with water for 2 sec. to show the prismatic dark interstitial material of unknown composition. Its presence in clinker is associated with the presence of Na_2O and K_2O.

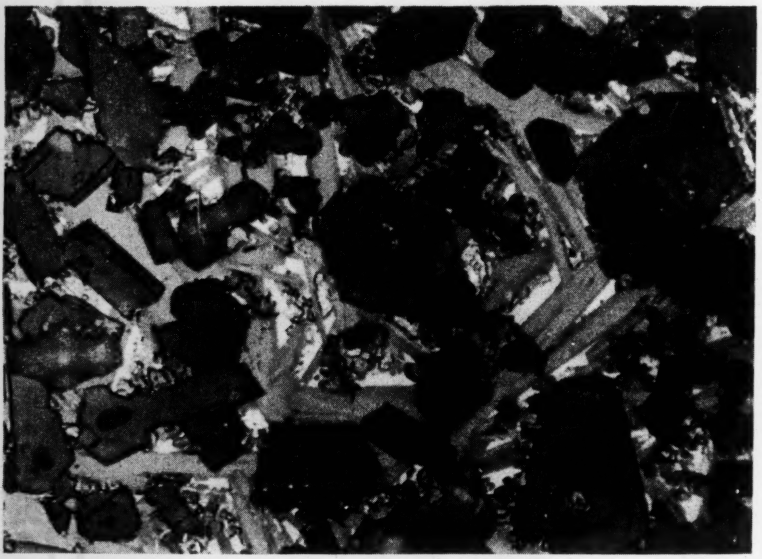

FIG. 6.—Same Area of Same Clinker as Fig. 5 Etched With Water for 2 secs. Followed by 1 per cent Alcoholic Solution of HNO_3 for 2 sec. (× 500).

Shows relation of interstitial material to C_3S crystals. C_3S crystals frequently have inclusions and border fringes of C_2S particles.

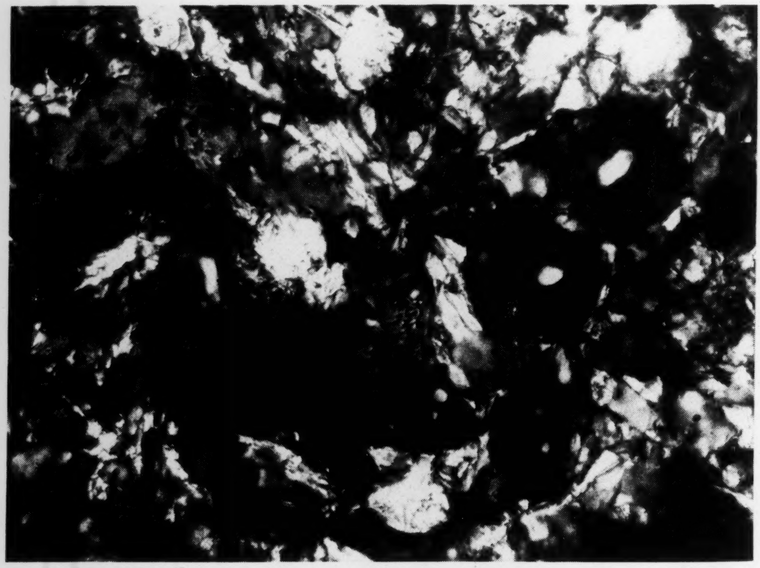

FIG. 7.—A Thin Section of a Normal Portland-Cement Clinker (× 500).

Between crossed nicols illuminated with transmitted light. The C_3S crystals are dark gray, the rounded C_2S crystals and the elongated C_4AF crystals are light gray. This illustrates the difficulty of distinction of constituents in photomicrographs with transmitted light.

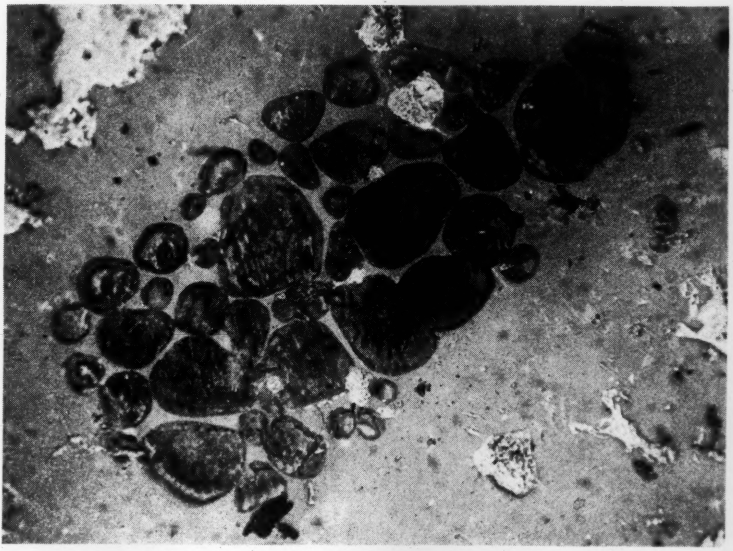

FIG. 8.—A Polished Section of a Normal Portland-Cement Clinker (× 1000).
Etched with 1:1 alcohol-water solution for 2 min. to show a cluster of rounded grains of free CaO.

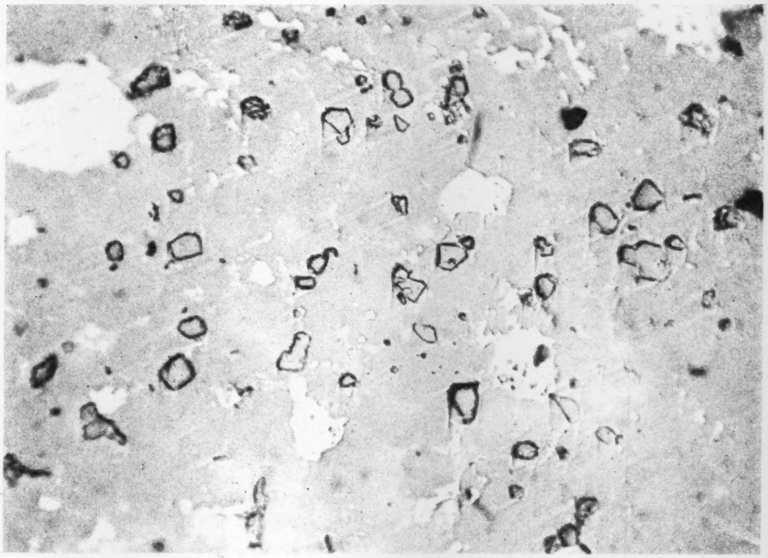

FIG. 9.—A Polished Section of a Normal Portland-Cement Clinker Showing Small Crystals of Free MgO (Periclase) Scattered Through the Clinker (× 500).

Figures 10 to 12 are the same polished *thin* section of a normal clinker examined in three ways to show the crystalline, doubly-refracting character of the prismatic dark interstitial material.

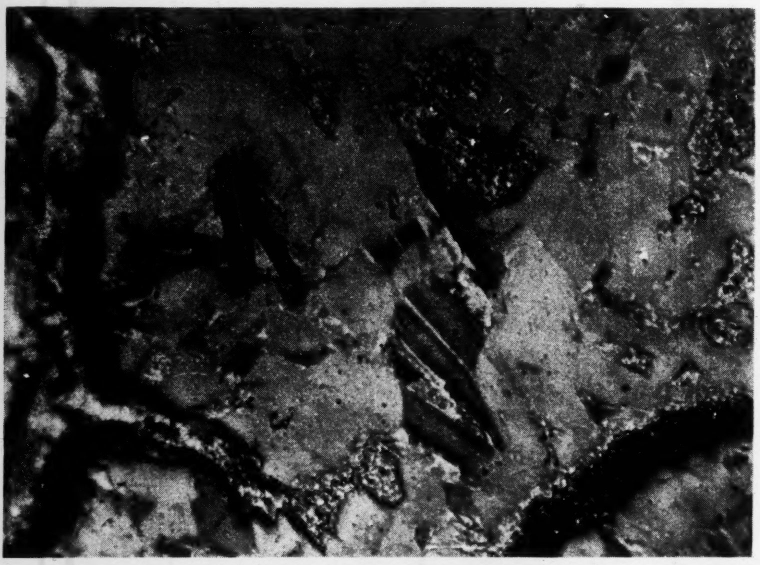

FIG. 10.—Normal Clinker Viewed with Reflected Light After Etching with Water for 2 sec. Two groups of prismatic crystals are shown near the center (× 500).

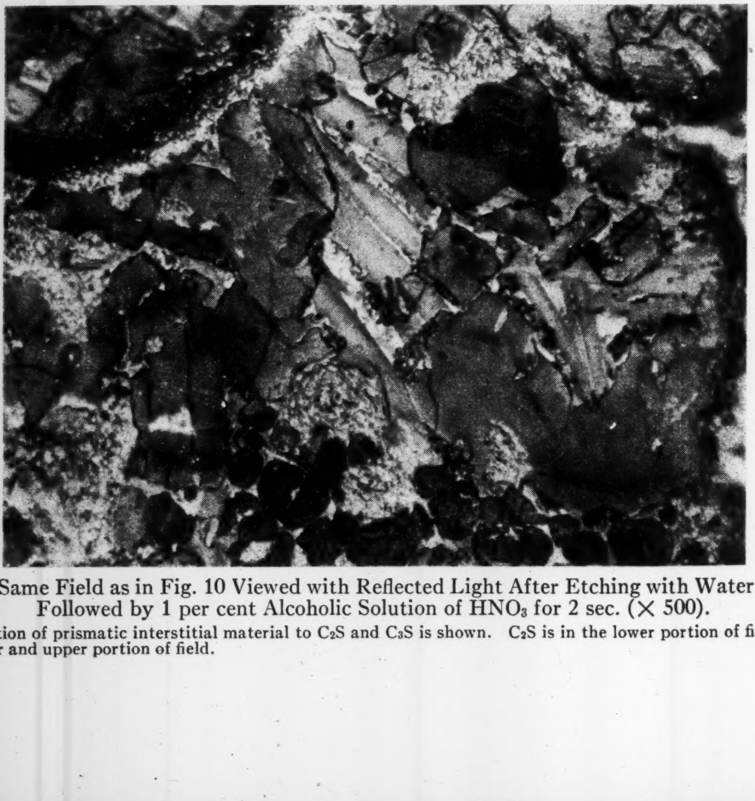

FIG. 11.—Same Field as in Fig. 10 Viewed with Reflected Light After Etching with Water for 2 sec. Followed by 1 per cent Alcoholic Solution of HNO_3 for 2 sec. (× 500).

The relation of prismatic interstitial material to C_2S and C_3S is shown. C_2S is in the lower portion of field and the C_3S in center and upper portion of field.

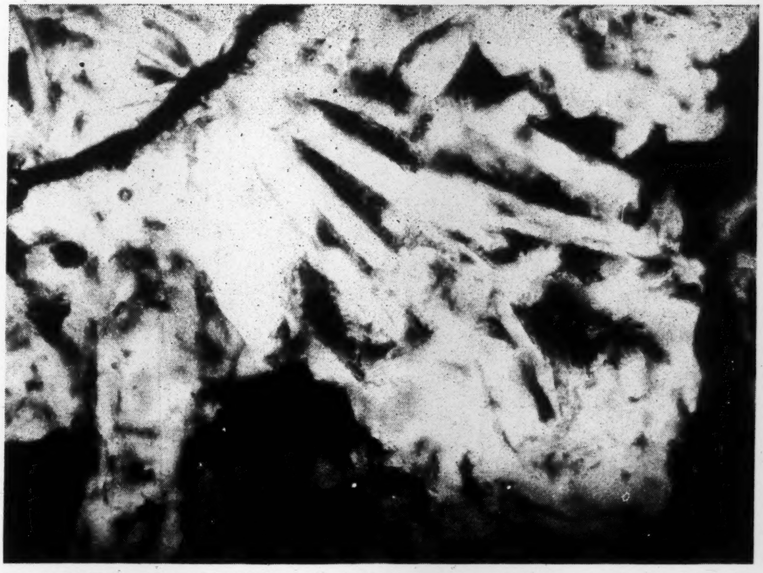

FIG. 12.—Same Field as in Fig. 10 Viewed with Transmitted Light and Crossed Nicols (× 500).

The fact that the prismatic material is light shows that it polarizes light and is, therefore, crystalline and doubly refracting.

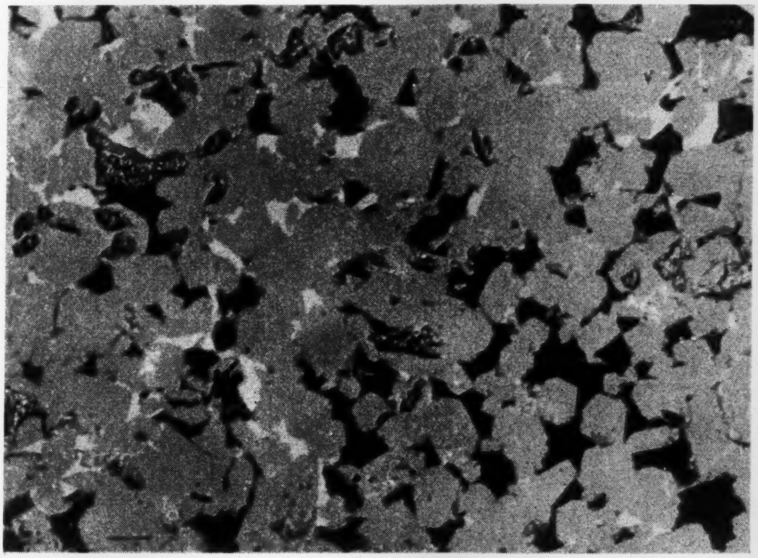

FIG. 13.—A Polished Section of a Laboratory-Prepared Clinker Containing No Na_2O or K_2O After Etching with Water for 2 sec. (× 500).

Note lack of prismatic character of dark interstitial material. The calculated compound composition is C_3S 55 per cent, C_2S 24 per cent, C_3A 12 per cent, C_4AF 9 per cent. The clinker had been heated at 1450 C. for 10 min., cooled from 1450 C. to 1250 C. at a rate of 10 deg. per minute and quenched from 1250 C. Compare with Figs. 14 and 15.

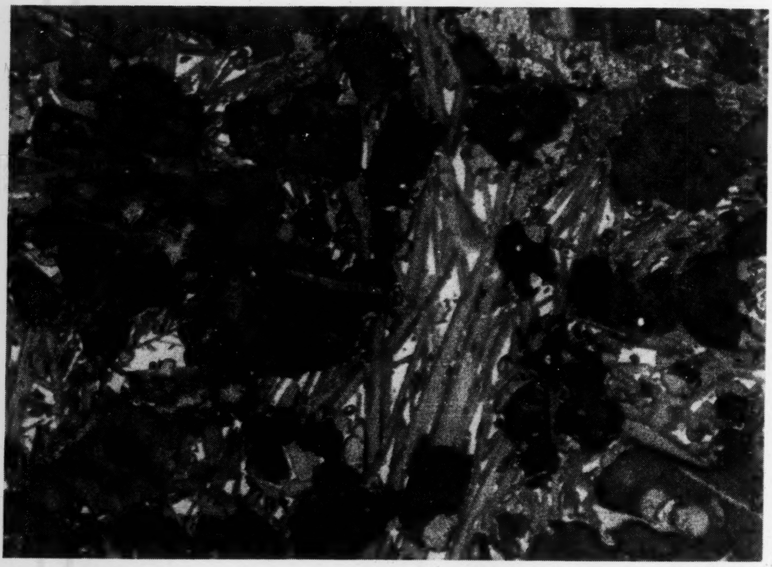

FIG. 14.—A Polished Section of a Laboratory Prepared Clinker of the Same Composition as That Shown in Fig. 13 to Which 1.5 per cent Na_2O Was Added Before Burning (× 500).

The same heat treatment was followed as with the sample illustrated in Fig. 13. Etched with water for 2 sec. followed by 1 per cent HNO_3 in alcohol for 2 sec. Note prismatic character of interstitial material.

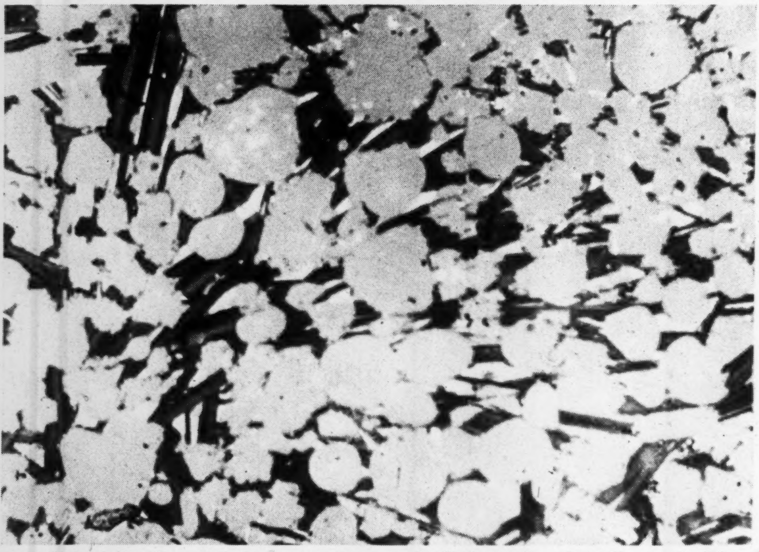

FIG. 15.—A Polished Section of Laboratory Prepared Clinker Like that Shown in Figs. 13 and 14, but 1.5 per cent K_2O Substituted for the Na_2O (× 500).

Etched with water for 2 sec. Note the prismatic character of interstitial material.

Figures 16 to 19, inclusive, are presented to illustrate the effect of "heat treating" clinker.

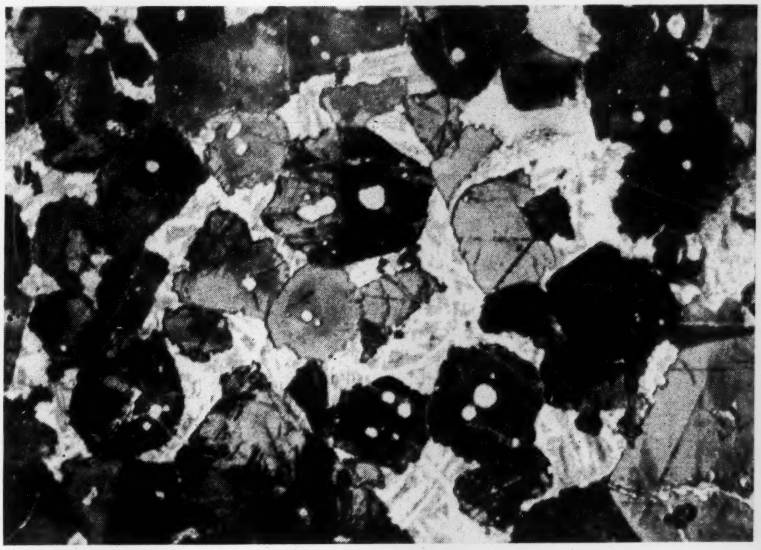

FIG. 16.—A Polished Section of Normal Clinker Etched with Water for 10 min. (× 500).
Note the sharp boundaries of C_3S crystals and contrast between light and dark interstitial material.

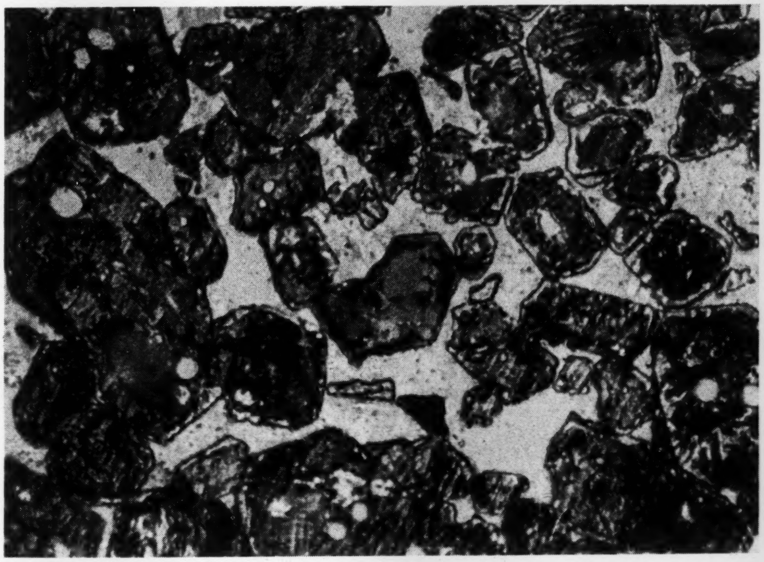

FIG. 17.—A Polished Section of "Heat-Treated" Clinker of the Same Composition and from the Same Mill as That Shown in Fig. 16 (× 500).
Etched with water for 10 min. Note definite border zone and heterogeneous character of most of the C_3S crystals. Light and dark interstitial material shows poor differentiation.

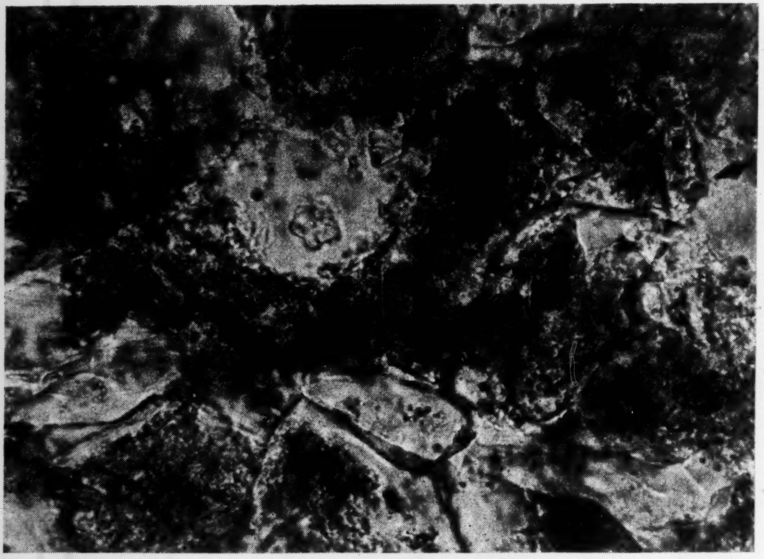

FIG. 18.—A Thin Section of the Same "Heat-Treated" Clinker as Fig. 17, Viewed in Ordinary Transmitted Light (× 500).

The iron-bearing interstitial material is in dispersed cloudy masses.

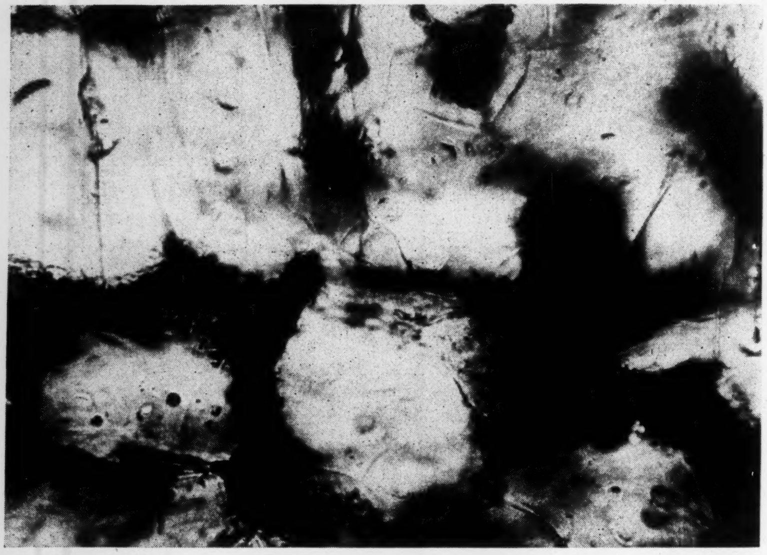

FIG. 19.—A Thin Section of the Same *Normal* Clinker as Fig. 16 (× 1000).

The iron-bearing interstitial material is in discrete groups in contrast with that in Fig. 18.

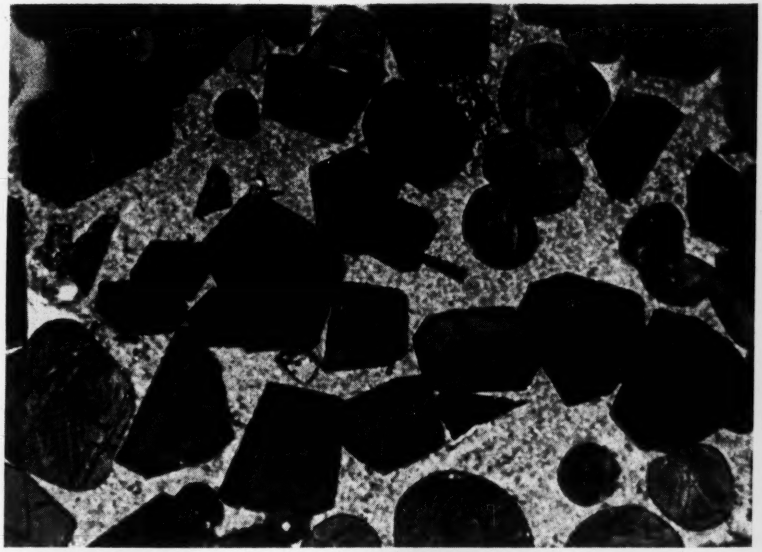

FIG. 20.—A Polished Section of a Normal Portland-Cement Clinker (Al_2O_3 to Fe_2O_3 Ratio = 1.78) (× 700).

Etched with water for 2 sec. followed by 5 per cent NH_4Cl for 3 sec. and 10 per cent KOH for 15 sec. With Al_2O_3 to Fe_2O_3 ratios of about this magnitude, the interstitial material does not tend to crystallize very well but gives a mottled appearance. The minute darker particles in the mottled interstitial material are glass.

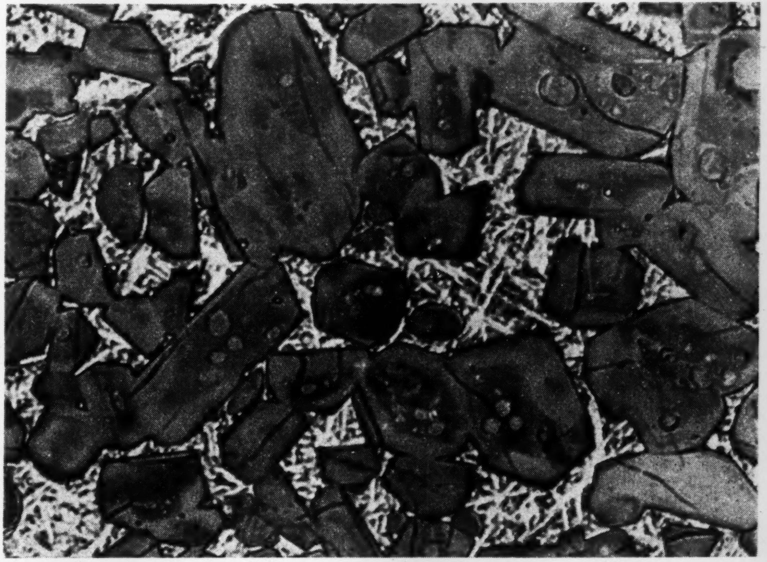

FIG. 21.—A Polished Section of a Moderate Heat of Hydration Clinker (Al_2O_3 to Fe_2O_3 Ratio = 1.34) (× 700).

Etched with water for 2 sec. followed by 1 per cent HNO_3 for 2 sec. and 10 per cent KOH for 15 sec. With Al_2O_3 to Fe_2O_3 ratios of this lower magnitude the interstitial material tends to crystallize. This is clearly shown in the figure. The glass (dark gray) is associated with prismatic crystals of C_4AF (light gray) in the interstitial material.

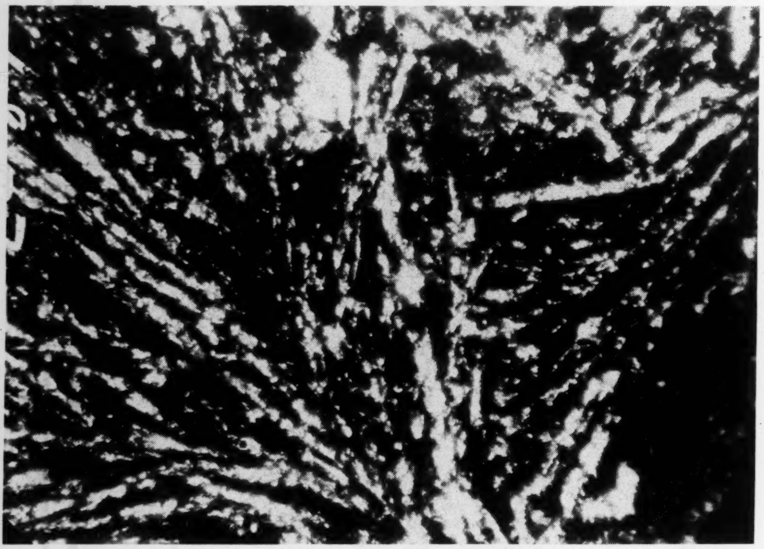

FIG. 22.—A Thin Section of Clinker Photographed by Transmitted Light (× 135).

The clinker and figure were prepared 26 yr. ago. The clinker was especially characterized by a SiO_2 and Al_2O_3 content of 18 and 11 per cent, respectively. At that time the technique of examination of etched sections by reflected light had not been developed. By the petrographic methods then in use, it was concluded that the formation appearing as elongated crystals was C_3S.

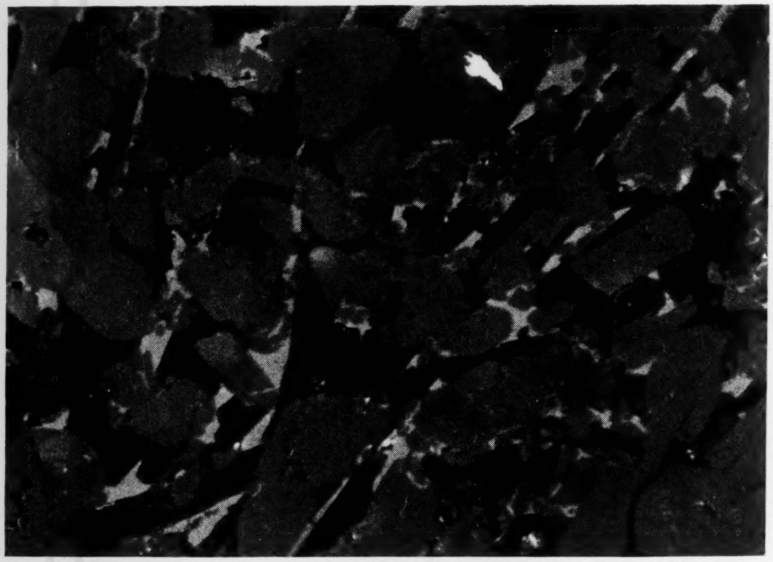

FIG. 23.—A Polished Section Very Recently Made of the 26 yr. Old Clinker Shown in Fig. 22 (× 750).

Etching with water reveals the elongated crystalline development to be the dark crystalline interstitial material (possibly C_3A in this case) noted in previous figures and not C_3S. The C_3S is not etched with water and appears in the figure as the gray angular crystals lying apparently on top of the elongated crystals.

SYMPOSIUM ON SIGNIFICANCE OF THE TENSION TEST OF METALS IN RELATION TO DESIGN

FOREWORD

By John M. Lessells[1]

Editor's Note.—The committee in charge of the Symposium on Significance of the Tension Test in Relation to Design, one of a series of technical discussions planned in connection with the subject "Significance of Mechanical Properties of Metals" consists, in addition to Mr. Lessells, *Chairman*, Massachusetts Institute of Technology, of G. F. Jenks, U. S. Army; H. F. Moore, University of Illinois; N. L. Mochel, Westinghouse Electric and Manufacturing Co.; R. E. Peterson, Westinghouse Electric and Manufacturing Co.; and R. L. Templin, Aluminum Company of America. The American Institute of Physics and American Society of Mechanical Engineers are cooperating closely. Mr. F. Seitz is acting as representative of the American Institute of Physics. Mr. Peterson is acting as representative of the A.S.M.E. Applied Mechanics Division.

The Symposium on the Significance of the Tension Test in Relation to Design is devoted to the presentation and discussion of a group of papers dealing with the tension test of metals, with the twofold object of attempting to clarify the relationships between fundamental characteristics measured in such a test and, in addition, the relating of such characteristics to the design problem.

The symposium considers only the tension test as made at normal temperatures and does not attempt to explore the effects of either high or low temperatures.

Forms of Tests Used

The various forms of tests used by the engineer to assess his materials can be divided into two groups: (*a*) fundamental type and (*b*) arbitrary type. The first type provides data which can be directly used for design purposes and the tension test is one of the most important, if not the most important, one in that group. The fatigue test is likewise an important test in this same group. The second type, or arbitrary tests, is well represented by the impact test and flexure test. While these are important tests in the control of materials they do not in their present stage provide data which can be directly used in design, hence their classification here as arbitrary. These facts have probably been recognized for the long period of years in which tension test data have been used. Even today many engineers continue to apply tension test data for the design of members subjected to a wide variety of stress conditions. Modern thought has probably contributed most to setting limits on the conditions where such data can be applied and has indicated those cases where more information is required for the best and most economical designs. It is of interest to observe here that from the results of modern research it is known that the fatigue strength is as nearly related to the tensile strength as with any other physical property. While this relationship varies over wide limits, designs subjected to cyclic or variable stress may be somewhat rational if based on the tensile strength. It is well known, however, that such an approach cannot be very accurate and it does not take into consideration the effect of stress concentration which can only be done by looking at the design problem from a wider viewpoint.

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[1] Chairman of Committee on Symposium on Significance of the Tension Test of Metals in Relation to Design; Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

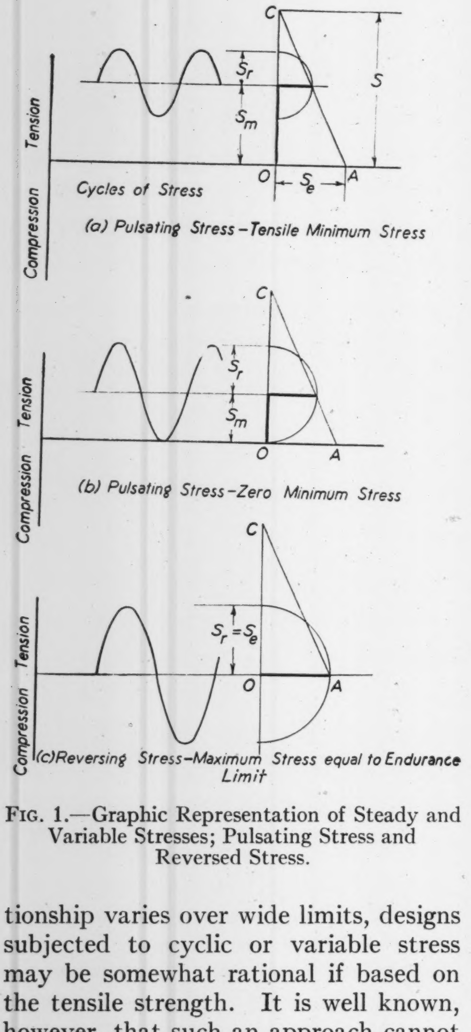
C
S_r
S
S_m
Tension
Compression
O
S_e
A
Cycles of Stress
(a) Pulsating Stress – Tensile Minimum Stress
C
S_r
S_m
Tension
Compression
O
A
(b) Pulsating Stress – Zero Minimum Stress
C
$S_r = S_e$
Tension
Compression
O
A
(c) Reversing Stress – Maximum Stress equal to Endurance Limit

Fig. 1.—Graphic Representation of Steady and Variable Stresses; Pulsating Stress and Reversed Stress.

Code for Working Stresses

In attempting to prepare a code for working stresses it is at once apparent that there are very few tests upon which a good foundation can be built. In the theory of elasticity, stress and strain are treated from a three-dimensional aspect but most of our knowledge concerning the behavior of materials rests on the results of simple unidirectional tests. In this respect the tension and fatigue tests are the most important. This picture may, of course, be changed when more data become available in the behavior of materials under combined stresses.

In the following the choice of working stress based on the yield strength or endurance limit is considered. It is recognized that in failure by buckling the critical stress rather than the yield strength is the governing factor. Such conditions have not to date been included in the code being discussed here.

Accepting the tension and fatigue tests as the basis of our reasoning, stresses are classified as either steady or variable. The generalized[2] condition of variable stress as in Fig. 1 (*a*) may be considered as consisting of a steady stress S_m and a variable stress S_r.

The case of a pulsating stress is represented in Fig. 1 (*b*) and a completely reversed stress by Fig. 1 (*c*). In this latter case $S_r = S_e =$ endurance limit for reversed stresses.

Materials are further classified as being either ductile or brittle. Obviously the limiting conditions for the case of steady stress must either be the tensile or the compressive strength and for that of variable stress the endurance limit.[3]

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[2] The following papers should be consulted: C. R. Soderberg, "Working Stresses," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 55, pp. 131-140 (1933). C. R. Soderberg, "Working Stresses," Design Data, Am. Soc. Mechanical Engrs. Schaubilder, "Dauerfestigkeit" *Zeitschrift* des Vereines deutscher Ingenieure (1933-1934).

Or if S = tensile or compressive strength
and S_e = endurance limit for reversed stresses
the variable stress $S_r \not> S_e$
and steady stress $S_m \not> S$

For the case of a ductile material the limiting condition will be as represented by *OAC* in Fig. 2. The line of failure for certain materials falls above *AC* but for simplification this is taken as *AC*. Such an assumption is, however, on the safe side. Any stress condition which falls outside of *OAC* according to this reasoning, which excludes hardening due to plastic yielding, will result in failure.

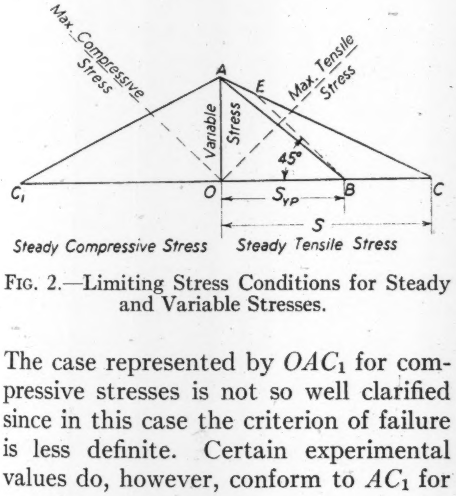

FIG. 2.—Limiting Stress Conditions for Steady and Variable Stresses.

The case represented by OAC_1 for compressive stresses is not so well clarified since in this case the criterion of failure is less definite. Certain experimental values do, however, conform to AC_1 for negative mean stresses.

In the case of steady stress for ductile materials it is customary to take not the value of the tensile strength $S = OC$ as the limiting stress but rather the value of the yield stress $S_{\text{y.p.}} = OB$. This means that the zone of failure is now represented by *OAB* for positive mean stresses. As before, any stress condition within this area will not result in failure and this area will represent all combinations of stress varying from a steady stress *OB* to a variable stress *OA*.

This general approach to the problem of working stresses has much to commend it. Some notable exceptions in the case of helical springs have been noted by Zimmerli[4] which deserve mention. Heat-treated chrome-vanadium steel springs to give a tensile strength of 205,000 psi. indicated a torsional endurance range of 75,000 psi. for stresses varying from zero to a maximum, whereas stainless steel spring (17.4 per cent chromium, 8.9 per cent nickel) treated to give a tensile strength of 216,000 psi. only indicated a torsional endurance range of 29,000 psi. when tested under similar stress conditions. Probably, however, these discrepancies, not explained by Zimmerli, are due to residual stress or surface effects which might be traced from a study of the elastic part of the tension test. In this discussion the effects of plastic flow due to tensile overstrain have been neglected. Haigh,[5] however, has shown that such overstrain can increase the maximum value of the variable stress to such a degree that the extreme value of $S_r + S_m$ approaches the tensile strength of the material. Some application of these ideas by Zimmerli to the improvement in helical springs by shot blasting will be mentioned later.

The above refers to ductile materials. For the case of brittle materials the tensile and compressive strength must be regarded as the limits of failure but this case is more complicated by the fact that the properties of such materials in tension and compression are usually different.

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[3] This assumes that the range of stress begins to decrease when the mean stress of the cycle becomes compressive. This is true in general but some exceptions have been noted.

[4] F. P. Zimmerli, "Permissible Stress Range for Small Helical Springs," *Bulletin 26*, Engineering Research Station, University of Michigan (1934).

[5] B. P. Haigh, "Fatigue in Structural Steel Plates with Riveted or Welded Joints," *Transactions*, Inst. Naval Architects, Vol. 81, pp. 84–116 (1939).

The above method allows also consideration being made of stress concentration effects. It is well known that rapid changes in section of machine parts lead to stress concentration. It is also generally accepted that for ductile materials such stress concentrations are less dangerous for cases of steady stress than for cases of variable stress. With this understanding, factors of stress concentration established by photoelastic research are well known and can be applied to the variable stress S_r (Fig. 1). The results obtained by the application of theoretical factors are, in cases of departure, on the safe side.

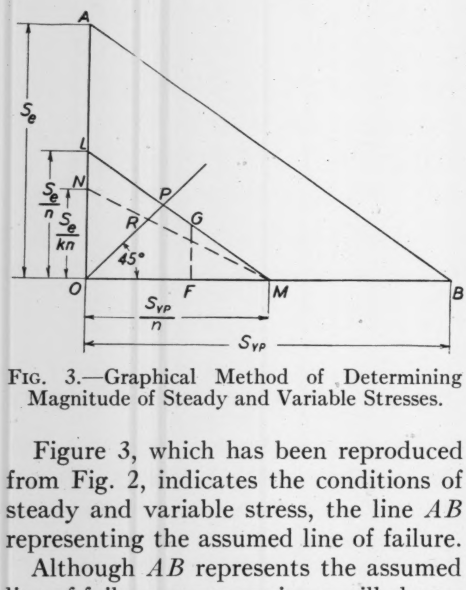

FIG. 3.—Graphical Method of Determining Magnitude of Steady and Variable Stresses.

Figure 3, which has been reproduced from Fig. 2, indicates the conditions of steady and variable stress, the line AB representing the assumed line of failure.

Although AB represents the assumed line of failure, every engineer will choose a factor of safety in order to cover the discrepancies which might occur in the load determinations, in the stress calculations, and in the material. If this factor be taken as n then the design limitations will be set by line LM where $OL = S_e/n$ and $OM = S_{y.p.}/n$. The general case[6] will then be determined by the coordinates of a point such as G where the steady stress S_m is represented by OF and the variable stress S_r by FG. Given the values of endurance limit and yield strength and a knowledge of the particular mode of stress variation the values of the variable stress S_r and the steady stress S_m can easily be obtained by graphical means.

If a machine part be made of medium-carbon steel such that the yield strength $(S_{y.p.}) = 42{,}000$ psi. and endurance limit $(S_e) = 30{,}000$ psi. and this part be subjected to a stress varying from 0 to $S_{\max.}$, then if the factor of safety n be taken as 2, triangles OAB and OLM can be drawn such that $OL = 30{,}000/2 = 15{,}000$ psi. and to the same scale $OM = 42{,}000/2 = 21{,}000$ psi. For the above stress condition $S_m = S_r$ and this will be represented by a line OP drawn from 0 at 45 deg. to OB. The coordinates of the point P of intersection of this line with LM gives the values of S_m and S_r. For this particular case $S_m = 8750$ psi. and $S_r = 8750$ psi. or $S_{\max.} = S_m + S_r = 17{,}500$ psi.

CONSIDERATION OF STRESS CONCENTRATION

If now this same machine part has a change in section such that the stress concentration factor[7] k is 1.4, the same approach can be used to arrive at a satisfactory design. For such conditions it is assumed that any correction for stress concentration need only be applied to the variable part of the stress. This assumption seems justified on the grounds that stress concentration when applied to steady stress on ductile materials will only lead to local yielding and hence a more favorable stress distribution.

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[6] *Loc. cit.*, C. R. Soderberg. See also S. Timoshenko, "Strength of Materials," Part II, D. Van Nostrand Co., New York, N. Y., from which the above example is taken.

[7] For tables of actual values see M. M. Frocht, "Factors of Stress Concentration," Design Data, Am. Soc. Mechanical Engrs. (1939); and N. C. Riggs and M. M. Frocht, "Strength of Materials," Ronald Press, New York, N. Y. (1938).

Applying these data to the case under consideration, point N is determined from the condition that $0N = S_e/kn = 30{,}000/1.4 \times 2$. The point N so obtained is joined with M and, as before, the coordinates of point R of intersection with the 45-deg. line from 0 determines the values of S_m as 7200 psi. and S_r as 7200 psi. This means $S_{\text{max.}}$ for this case becomes 14,400 psi. as compared with 17,500 psi. with no stress concentration.

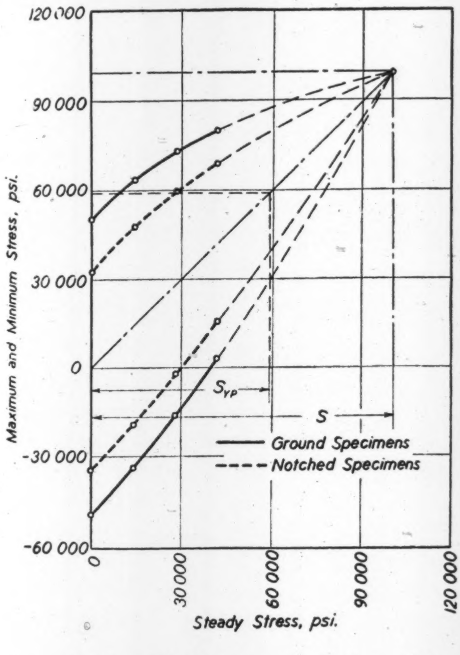

FIG. 4.—Graphical Method (Adopted by V.D.I.) for Determining Magnitude of Steady and Variable Stresses.

In other words this method of approach provides the designer with a tool whereby the effects of stress concentration can be fully regarded in his design.

Steady and variable stress conditions can also be represented by a diagram, the ordinates of which are maximum-minimum stress and the abscissas the mean stress of the cycle. This is the method shown in Fig. 4 as adopted by the Vereines deutscher Ingenieure[8] in their design sheets and has been found of considerable value in certain laboratories in this country. This method has the distinct advantage of showing at a glance the effects of stress concentration.

IMPORTANCE OF YIELD STRESS

The above brief summary of the present engineering viewpoint leaves many points on which more information is desired. In choosing the yield stress as the basis of failure for steady stress conditions are we on solid ground? This choice seems logical for those materials which have a definite yield point, but is the stress value corresponding to an extension of 0.2 per cent justified for materials like copper or aluminum alloys which have no yield point in view of the known benefits of plastic flow? The contributions of Nadai[9] and his coworkers have done much to clarify our understanding of yielding phenomena but much yet remains to be done. Since it is to be expected that work on combined stress will develop in future years, what should be the criterion of failure for such conditions? The beneficial effects of overstrain are mentioned above. From the work which has been done on the shot blasting of helical springs resulting in a very substantial increase[10] in the safe fatigue range under stress conditions which represent a steady torsional stress plus variable torsional stress, it may be that the choice of yield stress as now defined as the limit of failure for steady conditions is unduly conservative.

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[8] *Special Design Sheet No. 42*, V. D. I., October 21, 1933. Schenck, *Stahl und Eisen*, Vol. 52, p. 660. Also discussion by R. E. Peterson of Appendix to Report of the Research Committee on Fatigue of Metals: Nomenclature for Various Ranges in Stress in Fatigue, *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I, p. 162 (1937).

[9] A. Nadai, "Plasticity," McGraw-Hill Book Co., Inc., New York, N. Y ; also E. A. Davis, "Effect of Speed of Stretching and Rate of Loading on the Yielding of Mild Steel," *Transactions*, Vol. 60, pp. A-137–140, Am. Soc. Mechanical Engrs. (1938).

[10] A safe range of +20,000 psi. to +80,000 psi. was increased to +20,000 psi. to +120,000 psi. by shot blasting has been obtained by Zimmerli.

The importance which yield stress plays in the applications of materials may be shown in another way. Referring to Fig. 1 (*b*) it is apparent that if the stress remains pulsating and does not become reversing

$$S_m \gtreqless S_r$$

This means that a line *BE* drawn as in Fig. 2 at 45 deg. to *OC* will satisfy conditions that $S_m + S_r = S_{y.p.}$. Any point to the left of *E*, the point of intersection of fatigue line *CA*, will represent conditions where fatigue failure is likely to occur prior to yielding because

$$S_m + S_r \gtreqless S_{y.p.}$$

Any point to the right of *E* will represent conditions where yielding is likely to occur prior to fatigue because $S_m + S_r > S_{y.p.}$. In the case of an annealed 0.3 per cent carbon steel the value of S, $S_{y.p.}$, S_e, represented, respectively, by *OC*, *OB*, and *OA* will be of the order of 70,000, 37,000, and ± 30,000 psi., respectively. In the case of alloy steels the value of the yield stress $S_{y.p.}$ will be increased to a much greater percentage of the tensile strength than is the increase in endurance limit. For instance, for an oil hardened and tempered 3½ per cent nickel-chromium steel[11] the values of $S_{y.p.}$, S and S_e would be changed to 128,000, 138,000, and ± 68,000 psi., respectively. Accordingly, using the same convention, point *E* in Fig. 2 will move nearer to *C* increasing the possibilities of fatigue failures occurring before the yielding takes place.[12] It is recognized that point *E* is only a rough approximation since experiments show that fatigue failures may not take place until the stress has exceeded that at *E*. If, however, point *E* is taken, this means that in the case of a carbon steel there is more likelihood of local yielding occurring, thereby allowing a redistribution of stress, than in the case of alloy steels. To apply this to an actual design problem: is it better to use a carbon steel in an aircraft engine connecting rod stud where the yield point is nearer to the fatigue strength or an alloy steel where the yield point is considerably in excess of the fatigue strength?

HIGH YIELD RATIO STEELS

All of these phases of the strength problem seem to be involved in a discussion of strength properties as shown by the tension test. They also raise the question of the efficacy of high yield ratio steels under variable stress conditions.

IMPORTANCE OF DUCTILITY

Regarding the question of ductility values obtained from the tension test, engineers might classify ductile materials as those which show an elongation in the tension test of 5 per cent and over, and brittle materials as those which indicate less than 5 per cent. This is quite an arbitrary choice but obviously some line of demarcation between known ductile materials such as soft steel and copper and known brittle materials such as cast iron must be made in any rationalizing process. As regards the value of ductility which arbitrarily classifies the ductile and brittle materials, the engineer looks upon this as a safeguard against the effect of stress concentrations. Certain data,[13] although very meager, are available to support this viewpoint. The new ideas regarding ductility which we are now obtaining from previous work of Ludwik[14] and the more recent contributions of MacGregor[15] may help to clarify some of our ideas regarding the importance of ductility values. On certain points engineers are clear. It is generally understood that so-called ductile materials can be made to behave as brittle materials and that the usual sliding failure obtained on such can become substantially a separation failure. It may therefore be in order not to classify materials as brittle or ductile but as Nadai[16] has suggested refer to the brittle or the ductile state of materials.

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[11] H. F. Moore and J. B. Kommers, "An Investigation of Fatigue of Metals," *Bulletin 124*, University of Illinois (1921).

[12] This viewpoint is fully discussed in an article by B. P. Haigh, "Relative Safety of High Tensile and Alloy Steels Under Alternating and Pulsating Stresses," *Proceedings*, Inst. Automobile Engrs., Vol. 24, pp. 320–347 (1929).

[13] H. Fowler, "Solid Crank Axles," *Engineer*, December 5, 1924.

AIMS AND SCOPE OF SYMPOSIUM

It is for the purpose of bringing before the materials engineer the viewpoint of the designing engineer and to allow clarification of certain of the characteristics of the tension test some of which have been mentioned above, that the present symposium was organized.

The symposium consists of three papers dealing with the more fundamental aspects, the strength features, and the ductility features.

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[14] P. Ludwik, "Bruchgefahr und Materialprüfung," *Berichte 35*, Eidgenössische Materialprüfungsanstalt, Zürich (1928).

[15] C. W. MacGregor, "Relation Between Stress and Reduction in Area for Tensile Test of Metals," *Technical Publication 805*, Am. Inst. Mining and Metallurgical Engrs. (1937).

[16] *Loc. cit.*, Nadai; also *loc. cit.*, Timoshenko.

THE TENSION TEST

By C. W. MacGregor[1]

The short-time tension test is one of the most common methods used today to evaluate the physical properties of materials. Although experience has shown that the quantities determined in this manner are not in themselves sufficient to enable the engineer to predict the behavior of the material under all conditions of stressing, this form of experiment still constitutes the basic procedure in evaluating static material properties. Aside from the quantities determined directly, a wealth of practical experience has been built around the test, making it possible to predict, in many cases, satisfactory performance of the material from field experiences on materials having similar tensile properties. It has also been possible to use certain physical quantities, such as the yield strength determined under pure tension, together with modern theories of strength, in order to predict the behavior of metals under states of static combined stresses which occur so often in practice.

The tension test is usually conducted by applying increasing increments of axial load to a test bar, which may be either flat, round, or tubular, and by measuring the ensuing deformation with some form of strain gage. The procedure followed in this country for metals is covered in the A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8 - 36).[2] If the test bar is round as shown in Fig. 1, it has been the standard practice in this country to choose the diameter d_o of the bar equal to $\frac{1}{2} \pm 0.01$ in. and the gage length L_o equal to 2 ± 0.005 in. The choice of gage length and diameter of the specimen is usually different in each of the larger countries of the world necessitating certain conversion formulas if the final axial strains are to be compared. In recording the data, either load-deformation diagrams or stress-strain diagrams, referred to original dimensions, are plotted manually or by means of an autographic recorder.

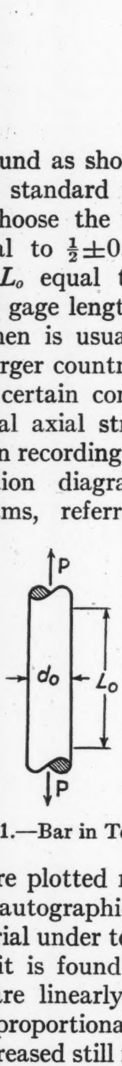

FIG. 1.—Bar in Tension.

If the material under test is mild steel, for example, it is found that load and deformation are linearly related up to the so-called proportional limit. When the load is increased still further, a value is reached at which a sudden instability in the equilibrium develops during which the load drops to a lower value and does not usually increase to its former magnitude until the test bar has elongated several per cent. The stress corresponding to the load, just before the sudden decrease, is known as the upper yield point and the stress corresponding to the lower value of the load after the drop-off, the lower yield point. The upper yield point is usually recorded. Following this period during which the deformation is not uniform along the test bar, the load begins to increase again on the testing machine, and the test bar lengthens uniformly, that is, with the exception of portions of the test bar near the enlarged ends the diameter of the bar for any given load is about the same along the length of the specimen. A maximum value of the load is soon reached, however, following which a local constriction appears somewhere along the length of the bar and the load again begins to drop off. The stress obtained by dividing the maximum load by the original area of cross-section is called the tensile strength and this stress value is always reported. After fracture the percentage elongation and the reduction of area are determined, the percentage elongation being computed by dividing the change in length by the original gage length and the reduction in area by dividing the change in area of cross-section by the original area.

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[1] Associate Professor of Applied Mechanics, Massachusetts Institute of Technology, Cambridge, Mass.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 750.

If the material happened to be one of the non-ferrous metals such as copper, the procedure would be much the same except that since no yield point is obtained, the so-called yield strength would be determined by the off-set method (A.S.T.M. Designation E 8 – 36).

The usual procedure then is to determine the proportional limit, the yield strength, the tensile strength, the percentage elongation, and the reduction of area, and if the stress-strain curve is plotted, this usually refers both the stress and the strain to the original dimensions of the test bar. This method has given us valuable information in the past, and will, no doubt, continue to do so in the future. That the method has certain limitations when looked at from a broader point of view has become apparent to many investigators. It is felt that at least one factor, which has in the past limited the true significance of certain tensile properties and perhaps curtailed their usefulness, especially in cases where the material is applied under conditions differing considerably from the test conditions, is that true physical stresses and strains are not determined. The effect of this is more pronounced, of course, in the plastic range near fracture. In order to correlate the stresses and strains determined in a tension test with those present in metal forming operations, cold-working processes, tests of materials subjected to combined stresses, and in general to attempt to clarify the laws governing the failure of materials under different conditions, a knowledge of true stress and strain values is essential. Stress and strain values based on the original dimensions of the test bar are not physically present except in the early stages of the stress-strain diagram, and it has little meaning to attempt to correlate them with those obtained in other processes. Further, the present methods used to depict ductility properties are subject to somewhat similar restrictions. In most of our specifications, both percentage elongation and reduction of area values are given. Attempts have been made to correlate these values with the general behavior of the material under service conditions. It would seem desirable to also determine true strains and reduction of area values for this purpose.

It is the author's feeling that much is yet to be learned about the tension test, and that while we have a lengthy background covering the routine tension test, we are not so fortunate with regard to experiments in which true stresses and strains are reported. Only later experiences will show whether values of stress and strain defined on a truly physical basis will yield more information in the application of our materials than the tests as ordinarily conducted. At present, it seems to be a safe assumption that such may prove to be the case. It is felt then that in order to discuss the physical significance of tension test values, the first step is to define these quantities in a physical manner and then to show the relationships obtained between them. In order to do this it will be necessary to depart from a consideration of the routine tension test. At present there is not enough experimental information available to relate some of these quantities to the behavior of our materials under service conditions not duplicated by those of the test. It is to be hoped that this gap in our knowledge will be filled in the near future.

In order that the statements which appear above may not be subject to misinterpretation, let it be stated here that for many routine tests, the procedures already in common use may still prove sufficient for the purposes for which they were intended. To obtain, however, a better physical picture of what the tension test really determines, and to correlate these values with those obtained in other tests and under other conditions of stressing, and to gain a better understanding of the general behavior of materials under stress, a certain departure from the customary testing procedures is necessary.

The subject of the tension test and its many ramifications might well fill at least one book of rather formidable proportions. It is therefore obviously not possible in such a short paper to discuss each phase in detail, and some subjects will of necessity have to be omitted. The paper, for the most part, will deal with the tension test on common engineering polycrystalline metals tested at normal speeds of testing and at normal temperatures. The following related subjects are intentionally omitted: tension impact; creep; short-time, high-temperature, tension tests on steels and normal-temperature tests on low-melting metals such as lead in which creep becomes a problem; the effects of heat treatment and other metallurgical questions; the so-called elastic portion of the stress-strain curve; hysteresis; experiments on single crystals; arbitrary definitions of yield strength; strength properties of the new high-strength alloy steels; aging; previous history of the metal before test; energy values; etc. Some of these questions will be discussed by others in the symposium. The intention is to give, first, a brief review of some of the important work done by various investigators in the past. This, it is hoped, will serve as a background for the subjects to follow. A discussion will then be made of some of the newer definitions of strain together with their relation to the strain values in common use, of ductility values defined in different ways as obtained from tests on various materials, and of true stress-strain curves constructed by different methods and based on recent experiments performed at the Massachusetts Institute of Technology.

PREVIOUS WORK

As was mentioned above, due to the very extensive literature existing on the tension test, space will permit reference to only a few of the important contributions on the subject. The following brief review does not, therefore, purport to be a complete résumé in any sense of the word, but is included to form a background for the succeeding discussion.

The stress-strain curve, or the load-elongation curve, obtained on a mild steel may be roughly divided into four parts: the so-called elastic region from zero load to the upper yield point, the yield point elongation portion, the region from the end of the yield point elongation to the maximum load point (the so-called uniform elongation portion), and the necking region during which the load on the testing machine drops off to fracture. For non-ferrous metals, the above divisions reduce to three in number. Many investigations have been carried out in which only one or two of these regions of the stress-strain curve were discussed. In the present review, each of these regions will be discussed with the possible exception of the elastic portion.

Stress-Strain Curves:

One of the most common methods of recording graphically the behavior of a metal subjected to tensile stress is to plot the tensile stress S_o, obtained by dividing the load P by the original area A_o, as a function of the strain ϵ_o, which is defined as the change in gage length ΔL_o from the original length L_o divided by the original length L_o. A list of the symbols used in the paper is given in an appendix to this paper.[3] While this procedure has proved of considerable value in the routine testing of materials, it does not have a very sound physical basis, as was first emphasized by Ludwik (**1**).[4] It has the disadvantage that a clear insight into the true physical behavior of the material when subjected to tensile stress is not evident. As was mentioned in a previous communication (**3**), some of the objections to this method are that: (1) a fictitious stress value is obtained which does not actually exist; (2) the strain ϵ_o becomes less and less exact for large values of ΔL_o; (3) when necking starts the strain varies considerably over the gage length and ϵ_o is not the true strain at any point on the outside of the bar; and (4) the axial strain varies over the cross-section of the necked portion, and ϵ_o does not represent the average of these strains over the cross-section.

To overcome some of these objections, various methods have been suggested. In some of these, only the stress is corrected by dividing the load by the actual area of the bar present when the load is acting (**4**), thus using the same definition of strain as mentioned above. Further, the stress so obtained is often called the *true stress* which, of course, is only the true stress as long as no actual necking has taken place. After necking has started, this value represents only the average of the true stresses acting in the axial direction (**5**). The method is still open to the objections 2, 3, and 4 listed above.

Ludwik (**1**) and later Hencky (**2**) suggested that the true strain be defined as

$$\epsilon = \int_{L_o}^{L} \frac{dL}{L} = \log \frac{L}{L_o}$$

in which case this definition holds for strains both large and small and approaches ϵ_o for small values of ΔL_o. The definition of strain given by Ludwik refers the change in gage length to the length from which that change was produced and not to the original length. According to this suggestion then $S = \frac{P}{A}$ would be plotted as a function of $\epsilon = \log \frac{L}{L_o}$. While this may be carried out as long as no necking takes place, as soon as the maximum load has been passed and a local constriction has begun and, if, as in the usual case, a gage length of 2 in. is used, the value of ϵ loses its physical significance, since it holds for a bar of uniform cross-section. To apply this method in the necking region of the bar, using a small gage length, certain practical difficulties are at once evident.

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[3] See p. 534.

[4] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this paper, see p. 531.

Stead (6) proposed that the average true stress $\frac{P}{A}$ be plotted as a function of the decreasing diameter. This procedure is free from certain of the disadvantages of the other methods mentioned and a convenient linear relationship is obtained between $\frac{P}{A}$ and d for the region of the curve from the value corresponding to the maximum load to fracture. The curve so obtained does not then have a strain value as abscissa, and is not applicable to flat bars.

A method in common use (7 to 14), especially in the deep-drawing industries, is to plot $S = \frac{P}{A}$ as a function of $q_o = \frac{A_o - A}{A_o}$ or the so-called reduction of area. Although this procedure has many advantages over previous methods of recording data, the value of q_o does not represent the true reduction of area, being subject to the same restrictions which apply to ϵ_o and hence not easy to correlate with true strain.

Norris (15) has obtained some interesting relationships by plotting the log of the true stress as a function of the log of the strain ϵ_o. It was found that a straight line was obtained for most materials throughout the major portion of the diagram.

A method was suggested recently (3) which has perhaps fewer disadvantages than former procedures designed to represent stress-strain data obtained from the tension test. According to the latter, the average true stress $S = \frac{P}{A}$ is plotted as a function of the true reduction of area $q' = \log \frac{A_o}{A}$. This will be described in some detail in the discussion to follow. During the preparation of this paper, it came to the author's attention that Maier (16) in Germany has also followed this same procedure independently. Since, however, only a few tests were reported and these were only incidental to the main portion of his experiments, Maier drew no conclusions as to the convenient linear portion of the diagram between the maximum load and fracture. Also no mention was made of different ways of constructing such curves, as, for example, the two-load method which will be discussed later on.

In certain problems it has been found worthwhile to plot curves of the octahedral shearing stress τ_n against octahedral shearing strain γ_n from data obtained by means of the tension test (17, 18).

Yield Strength and Speed Effects:

The phenomenon of the upper and lower yield points as exhibited by a mild steel is a fascinating subject and has incited the interest of a large number of experimenters (19 to 33). This is closely tied up with the formation of flow layers or the so-called Lüders' lines. The reasons for the discontinuous behavior with a sudden drop of the load at the yield point in a mild steel do not yet seem to be entirely clear. It has been shown by various investigators in recent years that this discontinuous behavior is not restricted to mild steels. A similar effect has been reported in zinc and cadmium crystals by Becker and Orowan (34); in duralumin and other aluminum alloys by Dawidenkow (35), Elam (36), Fell (27), and others; in brass by de Forest (37); in higher carbon steels, tungsten steel, and nickel steels at certain temperatures and straining speeds by Yuasa (38); and in single crystals of iron by Gensamer (31). Flow layers or Lüders' lines can, for example, be rather easily obtained in duralumin, and these layers will be identical in appearance to those obtained in mild steel. Some of the above materials, when tested with very precise equipment, showed for certain temperatures discontinuous yieldings during the entire yielding range to fracture, leading some investigators recently to the conclusion that possibly all plastic flow occurs in a stepwise discontinuous manner.

The effects of testing speed on the yield strength and the tensile strength have been investigated recently by Brinkman (**39**), Deutler (**40**), Elam (**36**), Winlock and Leiter (**41**), Davis (**42**), Morrison (**43**), Hougardy (**44**) and others. Davis conducted careful tests in which the rate of loading could be varied in the ratio of 21,000 to 1, independently of the rate of strain. He found, for example, that with a speed ratio of 14,300 to 1, the yield point of a mild steel increased about 29 per cent. Brinkman and Deutler varied the rate of strain by as much as 1,000,000 to 1. Their results checked Ludwik's logarithmic speed law: namely,

$$S = S_1 + S_2 \log \frac{v}{v_1}$$

where S_1, S_2, and v_1 are constants. It was found that the results reported by Winlock and Leiter also conformed fairly well to this law when the data were plotted on semi-logarithmic paper.[5] Winlock and Leiter showed also the importance of the grain size on the results obtained. For the steel tested by Deutler (20 per cent carbon), a speed ratio of 1000 to 1 increased the yield point 18 per cent, while the yield point of a steel (0.04 per cent carbon) investigated by Winlock and Leiter increased 55 per cent. For this same speed ratio, Deutler found an increase of 5.3 per cent in tensile strength, while Winlock and Leiter obtained an increase of 11.3 per cent. Morrison tested a low-carbon steel of the same carbon content as that used by Deutler and found a velocity effect slightly smaller although comparable in magnitude to Deutler's results. The above tests indicate that there may be a considerable difference in velocity sensitivity in the neighborhood of the yield point for different mild steels.

The effect of the speed of testing within the range which can be produced with ordinary testing machines has been investigated recently by H. F. Moore at the University of Illinois for the Society's Committee E-1 (Section on the Effect of Speed of Testing). It was found that although there is a distinct effect on tensile values, the effects are small and rather wide tolerances in the speed of testing can be permitted without introducing any serious error for most of the ductile metals. In some of the early experiments on $S - q'$ curves, which will be described later on, the author checked the general effect of the speed of testing on the quantities measured in the tension test and arrived at a similar conclusion, that is, that rather wide variations in the speed of testing produced only slight differences in either stress or strain values.

Austenitic steels have shown, on the contrary, rather large and erratic speed effects as was recently reported by Hougardy (**44**). In all cases the yield strength increased with the speed of testing. For some of these steels, the percentage elongation and reduction of area values increased with higher speeds of testing while in others these quantities decreased. The same result was found for the tensile strengths.

The Elongation Equation:

The problem of the conversion of percentage elongation data from one form of fractured tension test specimen to another has occupied the attention of numerous experimenters (**45** to **53**). The elongation equation provides the vehicle by means of which this may be accomplished. This equation has been established in many different forms, but in general it is made up of a term giving the local strain (which is not a true strain) plus another term which is the general or uniform strain. The need for such an equation arises from the fact that our specifications usually require the percentage elongation at fracture in 2 in. to be given, and the local strain portion of the equation depends on the ratio of the transverse dimension to the gage length. As was mentioned before, the percentage elongation value so measured is not a true strain, since the specimen is no longer of a uniform diameter and this quantity therefore has no clear physical meaning. It is felt that the need for such a conversion equation would not be evident if a different procedure were adopted in recording and defining ductility values. This will be discussed more fully later on.

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[5] This was done by one of the author's graduate students, F. Mittelberger.

Tensile Fracture:

When a ductile metal fails in tension, considerable local constriction takes place, accompanied by a three-dimensional stress distribution in the necked-down portion. The distribution of these stresses at fracture is not known. For certain metals, tests have shown that failure starts by means of a crack perpendicular to the axis of the specimen near the center, that is, at right angles to the direction of the average of the true stresses. There is reason to believe that the axial stresses present are larger than either radial or tangential stresses. Further, Maier (**16**) has shown that at fracture, tubes subjected to combined stresses produced by internal pressure and axial loading failed always when the principal stress reached a value equal to that obtained in a tension test by dividing the fracture load by the fractured area, and this independently of the combined stress ratio. These tests thus indicate an important application of the average of the true breaking stresses obtained by dividing the fracture load by the fractured area, that is, the possibility of using it to predict failure by fracture in metals subjected to combined static stresses. Only further experiments can check this point.

Morrison (**32**) has shown that the average of the true breaking stresses at fracture in tension is the same regardless of the state of the metal tested with reference to overstrain. He overstrained a mild steel in various fashions by combined tension and torsion, or tension only, and then tested in tension samples cut from these bars. Although the true ultimate tensile strength varied, the average of the true stresses at fracture remained the same for all cases. That such would be the case was predicted theoretically by Morrison on the basis that, at tensile fracture, the material consists of crystallites only, oriented at random, and the tensile stress to produce rupture should then be independent of the original state of overstrain in the material.

There is considerable evidence that the average breaking stress is more of a material constant than the true tensile strength. Theoretically, the true tensile strength is a point on the curve of true stresses and merely marks the position where local necking starts. The breaking stress is the maximum stress that the material is able to withstand in tension.

Ludwik (**54**) has shown that the breaking stress helps to bring out certain properties not reflected in changes in tensile strength such as effects of overheating, chemical influences, etc.

As was suggested by W. Kuntze (55) and discussed by Bain (56), Hoyt (57), McAdam and Clyne (58), and others, there is a need for information on the so-called "technical cohesive strength" of the original unstrained metal. The breaking stress for a necked-down tension specimen does not provide this information since the material has been cold worked. In order to obtain this value, Kuntze made tests on bars containing deep sharp notches which tended to restrain the metal from yielding, thus producing a separation type of failure. It has been shown that this technical cohesive strength first increases and then decreases with previous plastic deformation. The average true stress in a uniform test specimen continues to increase until failure by separation. The breaking stress in the tension specimen is then equal to the so-called technical cohesive strength for the cold-worked material.

STRAIN AND REDUCTION OF AREA

In order to record and specify the percentage change in dimensions of tension test specimens when subjected to stress, a number of definitions of strain have been suggested by engineers in the past. A few of these definitions will be discussed below.

It is perhaps only natural that the definition of strain which has come into the most common use in the testing of materials under uniaxial stress conditions should be essentially the same one which forms the basis of the strain analysis in the mathematical theory of elasticity. If the deformation ΔL_o, or the change in dimension, is small compared to the original dimension L_o from which this change takes place, then the strain by this theory is $\epsilon_o = \frac{\Delta L_o}{L_o}$. Since, for elastic conditions, the percentage change in L_o is very small, this length provides a convenient and obvious reference value upon which strain calculations may be based.

It has, however, become the custom today to use this convenient definition of strain for changes in length comparable in the order of magnitude to the original gage length. To mention a few examples of this, the percentage elongation values at fracture for various ductile polycrystalline metals tested in tension, the large deformations obtained with single crystals, the excessive deformations realized in many metal-forming operations which may be as much as 200 to 300 per cent (11), the deformations possible in rubber, etc., comprise only a few cases in which, although under large deformations, this same method of specifying strain has been used. In cases of this kind, some ambiguity exists in the choice of the proper reference length. No longer is the choice as apparent as it was for small deformations. Our intuitive conception of strain as a change in length referred to the length from which that change is produced leads us to the conclusion that, for deformations large or comparable in magnitude to the original gage length, our strain definition is more likely a summation process. It would then be desirable to derive such a strain expression which would be valid for deformations large or small.

In order to do this, let us consider a uniform tension test specimen of original gage length L_o and cross-sectional area A_o, and apply successively to it small increments of axial load ΔP_i. Corresponding to each step in the loading ΔP_i there will result small increments in length ΔL_i and decrements in area ΔA_i. Referring each small ΔL_i and ΔA_i to the length or area from which it was produced, as noted above, we obtain

$$P = \sum_{i=o}^{n} \Delta P_i, \qquad \epsilon = \sum_{i=o}^{n} \frac{\Delta L_i}{L_i},$$

$$q' = -\sum_{i=o}^{n} \frac{\Delta A_i}{A_i} \qquad \ldots(1)$$

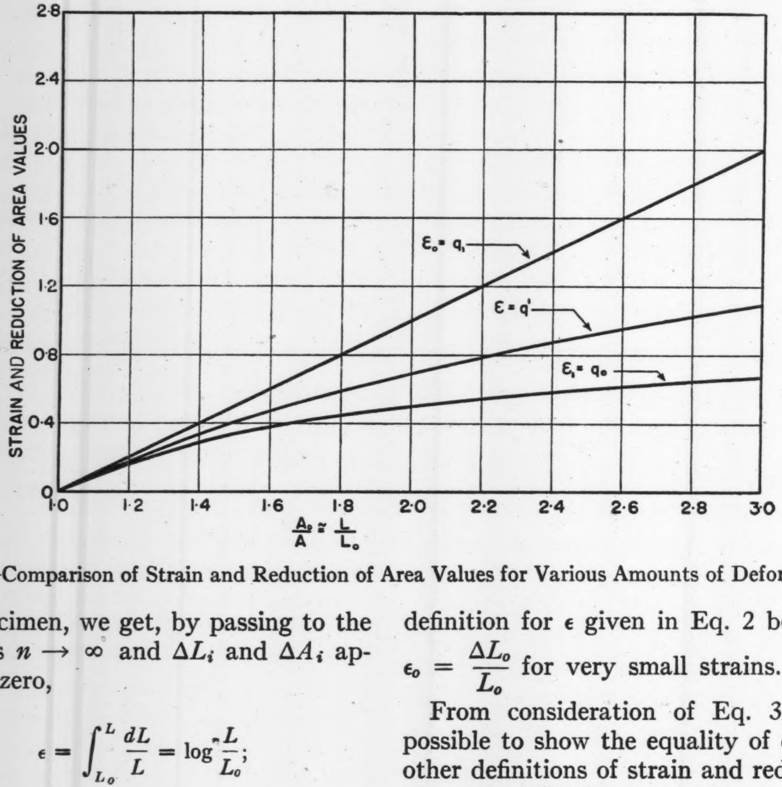

FIG. 2.—Comparison of Strain and Reduction of Area Values for Various Amounts of Deformation.

where P, ϵ, and q' are the total axial load, the true strain, and the true reduction of area, respectively. If L and A represent the final length and area of the specimen, we get, by passing to the limit as $n \rightarrow \infty$ and ΔL_i and ΔA_i approach zero,

$$\epsilon = \int_{L_o}^{L} \frac{dL}{L} = \log \frac{L}{L_o};$$

$$q' = -\int_{A_o}^{A} \frac{dA}{A} = \log \frac{A_o}{A} \qquad \ldots\ldots(2)$$

Since for most engineering ductile metals the volume remains constant when the material is plastically deformed (to within about 1 per cent error) within the ranges of deformation usually encountered in the tension test,

$$AL = A_o L_o \qquad \ldots\ldots\ldots\ldots(3)$$

and hence for a bar of uniform area of cross-section,

$$\epsilon = \log \frac{L}{L_o} = \log \frac{A_o}{A} = q' \qquad \ldots\ldots(4)$$

from which it is seen at once that the true strain and the true reduction of area are equal.

It can be shown that the true strain definition for ϵ given in Eq. 2 becomes $\epsilon_o = \frac{\Delta L_o}{L_o}$ for very small strains.

From consideration of Eq. 3, it is possible to show the equality of certain other definitions of strain and reduction of area which have been used in the past. For example, for a bar of uniform cross-sectional area,

$$\epsilon_1 = \frac{L - L_o}{L} = \frac{\Delta L_o}{L}$$

$$= q_o = \frac{A_o - A}{A_o} = \frac{\Delta A_o}{A_o} \qquad \ldots(5)$$

$$\epsilon_o = \frac{L - L_o}{L_o} = \frac{\Delta L_o}{L_o} = q_1 = \frac{A_o - A}{A} = \frac{\Delta A_o}{A} \quad \text{..(6)}$$

It is important to note that Eqs. 4, 5, and 6 are true only for specimens of uniform cross-sectional area, and do not hold when comparing strains, for example, over a 2-in. gage length of a fractured specimen, with the reductions in area. As will be discussed under the next heading, these equations can be used, however, when small enough gage lengths are chosen on fractured specimens.

From Eqs. 4, 5, and 6, the true strain ϵ and true reduction in area q' may be expressed in terms of the other values of strain or reduction of area given in the last two equations as follows:

$$\begin{aligned} \epsilon = q' &= -\log (1 - q_o) \\ &= -\log (1 - \epsilon_1) \\ &= \log (1 + \epsilon_o) = \log (1 + q_1) \quad \text{....(7)} \end{aligned}$$

Figure 2 shows the relative magnitudes of these different values of strain and reduction of area, plotted as a function of $\frac{A_o}{A}$ or $\frac{L}{L_o}$. It is seen that $\epsilon_o = q_1$ are always greater than the others; $\epsilon = q'$, or the true strain and reduction of area, values are next in magnitude, with $\epsilon_1 = q_o$, or the ordinary reduction of area, the smallest. Since we are accustomed to values of q_o, or the ordinary reduction of area, larger than the percentage elongation in 2 in. in the ordinary tension test, the relative values of these quantities might at first appear contradictory. That such is not the case is at once evident when it is realized that a comparison is made in Fig. 2 of values received on specimens of uniform cross-sectional area and not for the case of necked-down tension specimens where ϵ_o is over a large gage length including the constricted portion. The relative order of magnitudes given in Fig. 2 does, however, hold for strains over very short gage lengths even in the necked portion of the tension specimen after fracture.

It is possible to derive simplified expressions for the true strain ϵ and the true reduction of area q' which are often sufficiently accurate for the strain values usually encountered in polycrystalline ductile metals. This may be done by expanding the logarithm in a series as given in Eq. 8.

$$q' = \log \frac{A_o}{A} = 2\left[\frac{\frac{A_o}{A} - 1}{\frac{A_o}{A} + 1} + \frac{1}{3}\left(\frac{\frac{A_o}{A} - 1}{\frac{A_o}{A} + 1}\right)^3 + \frac{1}{5}\left(\frac{\frac{A_o}{A} - 1}{\frac{A_o}{A} + 1}\right)^5 + \cdots \right] \cong \frac{\Delta A_o}{\frac{A_o + A}{2}} \quad \text{..(8)}$$

The terms beyond the first in the above series are usually very small as compared to the initial term, since the series is rapidly convergent, and can therefore be neglected. Hence, the true reduction of area is essentially the change in area divided by the average of the initial and final areas.

This same procedure may be carried out for the true strain ϵ, and making use of Eq. 7 we may then derive the following approximate expressions for the true strain, or true reduction in area:

$$\begin{aligned} q' = \epsilon &\cong \frac{\Delta A_o}{\frac{A_o + A}{2}} = \frac{\Delta L_o}{\frac{L_o + L}{2}} = \frac{\epsilon_o}{1 + \frac{\epsilon_o}{2}} \\ &= \frac{q_1}{1 + \frac{q_1}{2}} = \frac{q_o}{1 - \frac{q_o}{2}} = \frac{\epsilon_1}{1 - \frac{\epsilon_1}{2}} \cong \frac{\Delta d_o}{\frac{d_o + d}{4}} \quad \text{..(9)} \end{aligned}$$

In the last term of Eq. 9, the true strain is given in terms of the initial diameter d_o and the final diameter d of a round bar.

A comparison is made in Table I of the magnitudes of strain and reduction in area given by these different definitions. It may be seen that corresponding to a strain ϵ_o of 50 per cent, the approximate expressions given in column 5 are accurate to within about 1 per cent as compared to the true values given in column 4. For a strain ϵ_o of 100 per cent, the approximate values in column 5 deviate by 3.9 per cent from those in column 4. For strains greater than these, the approximate expressions are too much in error.

TABLE I.—COMPARISON OF STRAIN AND REDUCTION OF AREA MAGNITUDES.

$\frac{A_o}{A} = \frac{L}{L_o}$	$q_1 = \frac{A_o - A}{A}$ $\parallel$ $\epsilon_o = \frac{\Delta L_o}{L_o}$	$\epsilon_1 = \frac{L - L_o}{L}$ $\parallel$ $q_o = \frac{\Delta A_o}{A_o}$	$\epsilon = \log \frac{L}{L_o}$ $\parallel$ $q' = \log \frac{A_o}{A}$	$\epsilon \cong \frac{\Delta L_o}{\frac{L + L_o}{2}}$ $\parallel$ $q' \cong \frac{\Delta A_o}{\frac{A_o + A}{2}}$
1	*2*	*3*	*4*	*5*
1.001	0.001	0.0009990	0.0009995	0.0009995
1.010	0.010	0.00990	0.00995	0.00995
1.100	0.100	0.0909	0.09531	0.0952
1.200	0.200	0.1668	0.1823	0.182
1.300	0.300	0.231	0.2624	0.261
1.400	0.400	0.286	0.3365	0.334
1.500	0.500	0.334	0.4055	0.400
2.000	1.000	0.500	0.6932	0.666
3.00	2.000	0.666	1.0986	1.000
10.00	9.000	0.900	2.302	1.635

Referring to $\epsilon_o = 50$ per cent again and comparing columns 2 and 4 it may be seen that ϵ_o is different from ϵ by 23 per cent. The divergence between the two, of course, widens with larger strain values as shown.

DUCTILITY VALUES

In the past, various measures of ductility have been proposed (**59**). It is rather common practice today to consider the percentage elongation in 2 in. ϵ_o together with the percentage reduction of area as convenient ductility values for round specimens. It is recognized that both of these quantities give some information not included in the other, although there is a tendency in various quarters to favor the reduction of area as perhaps being the more reliable ductility guide of the two (**60, 61**). While figures on reduction of area are always more difficult to obtain on flat specimens, this can be done reasonably well if a standard size specimen is used. In case sheets of different thicknesses are tested, this is not the case and reliable figures on reduction of area are not easy to obtain. In order to have better ductility guides for sheet material, various forms of tests have been devised ranging from the wedge-drawing apparatus of Sachs (**62**) to attempts to simulate the deep-drawing applications by means of the Ericson cupping test, and others of a similar kind. As Templin (**59**) points out, there is considerable lack of agreement in the results obtained by these different forms of test on sheet metals.

It is the purpose herein to confine the discussion, therefore, to ductility values obtained on round bars. Proposals have been made at various times to use the uniform elongation, that is, the elongation in mild steel from the yield point to the maximum load, as an additional measure of ductility. This would have the advantage that it is independent of the size of the test specimen. The idea has been usually to obtain this uniform elongation from axial strain measurements.

Let us then examine the possibility of using diameter measurements to obtain a more exact determination of ductility values. If simultaneous measurements of load and diameter are made during the test, the diameter corresponding to the maximum load can easily be obtained and the diameter just before yielding starts is also known.

This latter diameter can be taken as the original value, since the difference can hardly be noted on a micrometer or dial gage and clamp, whichever instrument is used. The true strain ϵ_u or the true reduction of area q'_u at the maximum load (which is the true uniform strain) is then

$$q'_u = \epsilon_u = \log \frac{A_o}{A_u} \quad \ldots\ldots\ldots (10)$$

where A_o is the original area and A_u that at the maximum load. When the test specimen necks down and fractures, the true strain ϵ_b or the true reduction of area q'_b at fracture is

$$q'_b = \epsilon_b = \log \frac{A_o}{A_b} \quad \ldots\ldots\ldots (11)$$

where A_b is the fracture area. Since both A_u and A_b are known, the true local necking strain ϵ_n or true local necking reduction of area q'_n becomes

$$q'_n = \epsilon_n = \log \frac{A_u}{A_b} \quad \ldots\ldots\ldots (12)$$

It is then readily seen that

$$q'_b = q'_u + q'_n \quad \ldots\ldots\ldots (13)$$

or that

$$\epsilon_b = \epsilon_u + \epsilon_n \quad \ldots\ldots\ldots\ldots (14)$$

In other words the strains when defined in this way add directly. *That is, the true strain or reduction of area at fracture is equal to the direct sum of the true uniform strain and the true local necking strain.* When defined by the older ϵ_o method, such superposition is not possible even if the bars remained of uniform diameter. If the approximate definitions of ϵ and q' given in Eq. 9 are used, it can also be demonstrated that Eqs. 13 and 14 are also approximately true. This serves to bring out one additional advantage of the logarithmic definition of strain.

With regard to ductility values for the tension test, then, it is proposed herein that two new quantities be used to define such ductility, namely $\epsilon_u = q'_u$ and $\epsilon_n = q'_n$ in place of the commonly reported values of ϵ_o and q_o. Since neither ϵ_u nor ϵ_n depend on a fixed gage length, comparisons of ductility values obtained on bars of different sizes may easily be made without the necessity of using the elongation equation. In fact, by following the above definitions of strain, the need for a complicated elongation equation of conversion would no longer be evident. The ϵ_o value determined over a 2-in. gage length would not represent the true strain even if the specimen remained uniform in cross-sectional area, and since local necking takes place, the strain varies widely throughout this length. Further, the value of q_o does not represent a true reduction of area and is not conveniently related to a true strain. The true local necking strain ϵ_n is determined from its equality with the true local reduction of area q'_n, which equality assumes constancy of volume. That constancy of volume holds to within a small percentage error has been demonstrated frequently. The actual gage lengths involved in the definition of ϵ_n are very small, and instead of computing ϵ_n from these, the convenient procedure is to determine it from q'_n, its equivalent obtained by measurements of the diameter. The value of ϵ_u gives the true uniform strain before local necking starts, ϵ_n is the additional true strain produced through local necking, and the sum of the two ϵ_b represents the true maximum strain at the bottom of the neck which the material is capable of withstanding before fracture. It would appear then, that the values of ϵ_u and ϵ_b found in this way may give more information regarding the deformation characteristics of the material than ϵ_o and q_o.

The experimental technique involved in accomplishing this result is indeed simple and need be no more complicated than that used in the measurement of ϵ_o and q_o. The only equipment necessary is a dial gage and clamp to measure the diameters which are determined corresponding to each load reading. Indeed, axial strain measurements need not be taken.

TABLE II.—COMPARISON OF DUCTILITY VALUES AT MAXIMUM LOAD AND AT FRACTURE FOR VARIOUS MATERIALS SUBJECTED TO TENSILE STRESS.

At Fracture				At Maximum Load		Material and Heat Treatment
$\epsilon_o = \frac{\Delta L_o}{L_o}$ in 2 in.	$q_b = \frac{A_o - A_b}{A_o}$	ϵ_n or $q'_n = \log \frac{A_u}{A_b}$	ϵ_b or $q'_b = \log \frac{A_o}{A_b}$	ϵ_u or $q'_u = \log \frac{A_o}{A_u}$	$q_u = \frac{A_o - A_u}{A_o}$	
0.309	0.516	0.540	0.727	0.187	0.170	VD 95 per cent Carbon Steel. Specimen NB. As Received.
0.365	0.606	0.719	0.932	0.213	0.191	S.A.E. 1112 Steel. Specimen 7A. Annealed at 1650 F. for 1 hr. Furnace cooled.
0.150	0.464	0.552	0.632	0.0797	0.0776	S.A.E. 1112 Steel. Specimen 7B. As received cold worked state.
0.433	0.730	1.045	1.32	0.275	0.240	Boiler Rivet Steel. Specimen 2B. Annealed at 1700 F. for 1 hr. No pre-strain.
0.330	0.657	0.870	1.07	0.199	0.180	Boiler Rivet Steel. Specimen 2E. Pre-strained 13 per cent and Aged at 212 F. for 1 hr.
0.334	0.586	0.724	0.891	0.167	0.148	Mild Steel. Specimen 3A. Pre-strained 1.86 per cent. Aged at 212 F. for 1 hr.
0.288	0.563	0.706	0.832	0.126	0.115	Mild Steel. Specimen 3C. Pre-strained 4.50 per cent and Aged as above.
0.224	0.580	0.778	0.871	0.0925	0.0847	Mild Steel. Specimen 3B. Pre-strained 8.42 per cent and Aged as above.
0.250	0.452	0.415	0.602	0.187	0.171	Non-Heat-Treated Duralumin. Specimen 3.
0.447	0.815	1.46	1.69	0.230	0.206	Commercially Pure Aluminum. Specimen K. Annealed at 700 F. for 4 hr.
0.179	0.671	1.09	1.12	0.0278	0.0274	Commercially Pure Aluminum. Specimen M. As Received.
0.413	0.348	0.081	0.425	0.344	0.292	Cold-Drawn Brass 65-35. Specimen E. Annealed at 800 F. for 2 hr.
0.218	0.465	0.477	0.627	0.150	0.139	Cold-Drawn Brass 65-35. Specimen F. As Received.
0.601	0.754	1.06	1.40	0.343	0.289	Commercially Pure Copper. Specimen A. Annealed at 800 F. for 2 hr.
0.149	0.605	0.921	0.933	0.0119	0.0119	Commercially Pure Copper. Specimen C. As Received.

In order to indicate the relative magnitudes of some of these ductility values, tension tests were made recently on various metals at the Massachusetts Institute of Technology. Table II shows a comparison of the strain and reduction of area magnitudes obtained at the maximum load and at fracture. The first two columns give the values of strain ϵ_o in 2-in. and the reduction of area q_o which are usually reported. It will be noted that, in every case, the values of true uniform strain ϵ_u and true local strain ϵ_n yield more information on the deformation characteristics of the material than ϵ_o and q_b given in the first two columns. For example, let us compare the strain values for the case of the commercially pure copper in the as-received state, test specimen C. The ratio of $\frac{q_b}{\epsilon_o} = 4.06$, while $\frac{\epsilon_n}{\epsilon_u} = 77.5$. The latter ratio shows that there existed very little uniform strain with an excessive local strain. On the other hand, let us compare the strain values for the annealed brass, test specimen E. The inverse ratio of $\frac{\epsilon_o}{q_b} = 1.19$, while $\frac{\epsilon_u}{\epsilon_n} = 4.26$. Although the effect is in the same direction, the latter ratio shows in perhaps a clearer way that this material exhibits a uniform strain several times larger than the local necking strain. A similar effect will be noted in the other test values shown. The fact that ϵ_o includes both local and general strain which vary along the length, clouds up the picture of deformation.

Another interesting comparison may be drawn from Table II. The fourth column gives values of ϵ_b, or the maximum true axial strain which the material may withstand before fracture. The value of ϵ_b varied from 1.03 to 6.27 times ϵ_o for the different materials tested. In other words, in cases such as represented by test specimen C of commercially pure copper in the as received condition, where excessive local necking strain takes place without appreciable uniform strain, the true axial strain becomes 6.27 times the ϵ_o in 2 in. On the other hand, if very little local strain develops as for annealed brass, the values are close together.

The effect of strain-aging a mild steel in increasing the ratio of local strain to general or uniform strain is also pictured in a somewhat better fashion by the ϵ_u and ϵ_n values in the table as evidenced by the mild steel test specimens 3A, 3B, and 3C.

A number of the test specimens included in Table II were ruled every 0.1 in. with very fine circumferential scratches produced by means of a diamond tool. A.S.T.M. standard 0.505-in. diameter test specimens were used, and before the test the diameters were carefully measured at each scratch and the distances between the scratches also determined with a comparator, accurate to 0.0001 in. These specimens were then tested in a 30-ton hydraulic testing machine, and the loads and minimum diameters read simultaneously throughout each run. After fracture, the diameters along the bars and the distances between the scratches were again determined and the values of ϵ_o, $\epsilon = q'$, and q_o computed for each division. The results were then plotted along the axis of the bar.

Figures 3 to 9 show the variation of these strain values along the length of each test specimen in the final, as-fractured position. Several materials were chosen, in various conditions of cold work, to show the differences obtained. It will be noted that for each figure the ϵ_o values were the greatest for every position along the bar, the $\epsilon = q'$ next in order of magnitude, and the q_o values the smallest. The values of ϵ are plotted as open circles, while those of q' are indicated as solid circles. It can be observed that they are equal everywhere along the test bars, since they follow the same curve intermediate between the curves of ϵ_o and q_o. These tests then show that we are justified in equating ϵ and q', that is, the true strain and true reduction in area, respectively.

The differences in the deformation characteristics, under tension, of annealed and unannealed metal can be seen by comparing Fig. 3 with Fig. 4 for S.A.E. 1112 steel, Fig. 5 with Fig. 6 for aluminum, and Fig. 7 with Fig. 8 for brass. The annealing procedure followed is given in Table II for each material. It is apparent that the annealing treatment was more effective in increasing the ductility of the metal not located immediately in the region of local constriction than it was in increasing the strain values in the necked-down portion. Fig. 8 shows how entirely different the mechanism of de-

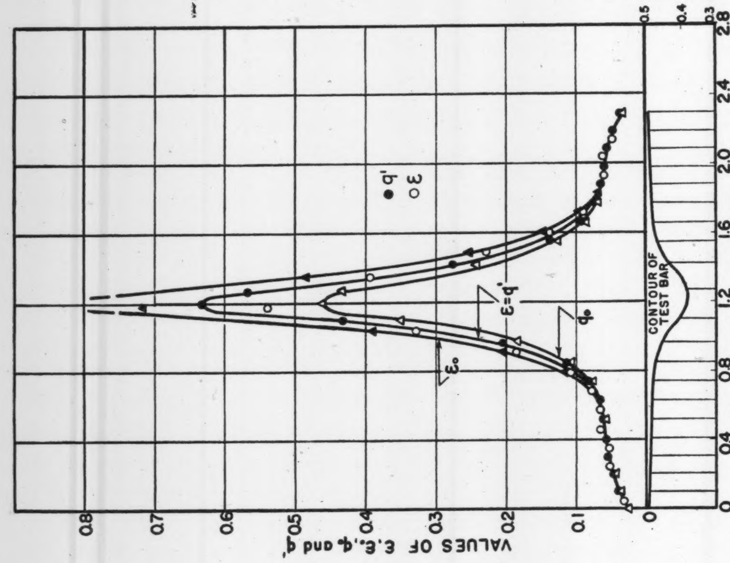

FIG. 3.—Strain and Reduction of Area Along Fractured Test Specimen 7B of S.A.E. 1112 Steel (as Received).

NOTE.—Refer to Table II for details of ϵ_0 in 2 in. and other data.

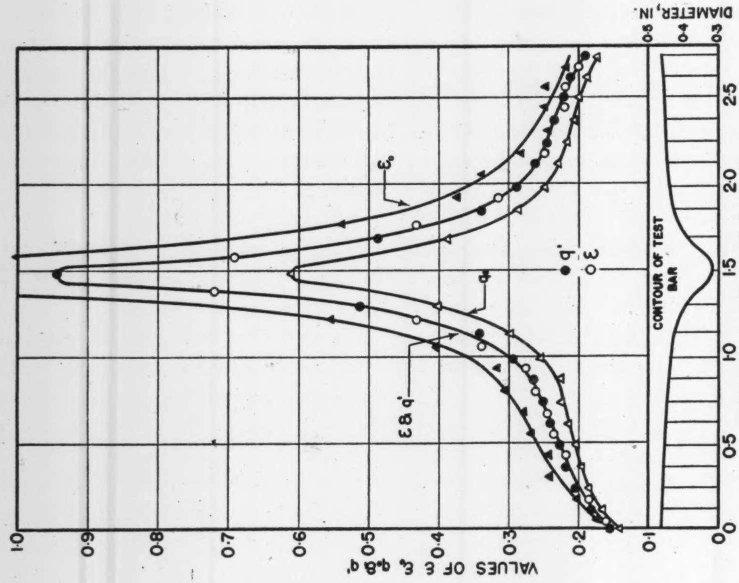

FIG. 4.—Strain and Reduction of Area Along Fractured Test Specimen 7B of Annealed S.A.E. 1112 Steel.

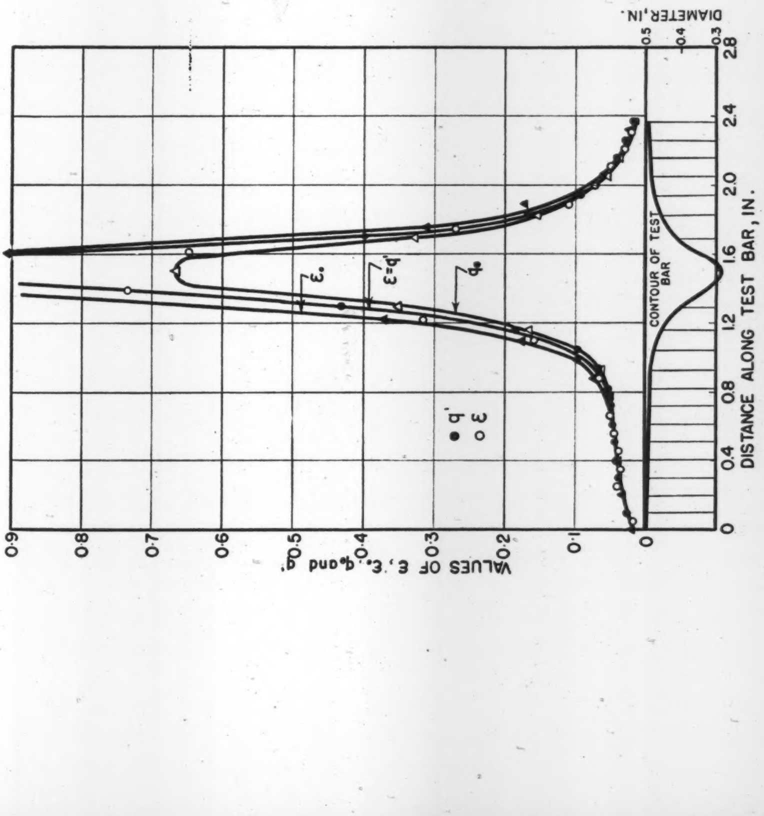

Fig. 5.—Strain and Reduction of Area Along Fractured Test Specimen M of Commercially Pure Aluminum (as Received).

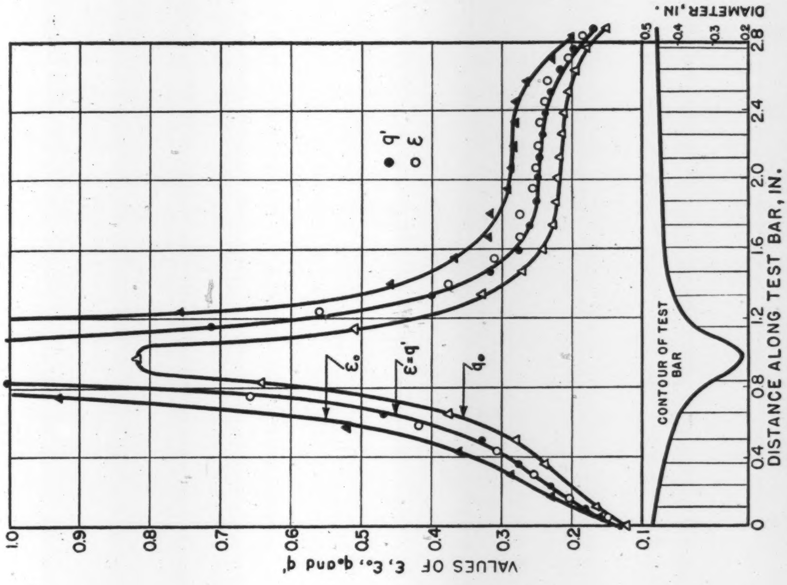

Fig. 6.—Variation of Strain and Reduction of Area Values Along Broken Test Specimen K of Annealed Pure Aluminum.

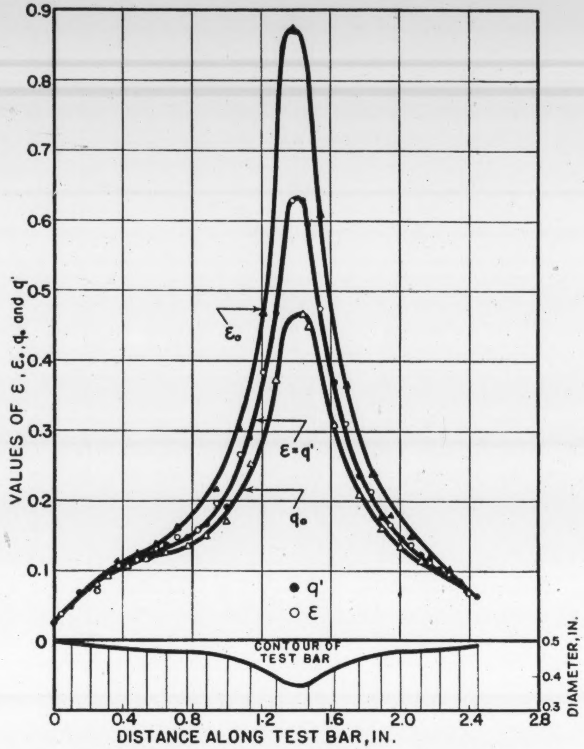

FIG. 7.—Variation of $\epsilon = q'$, ϵ_0 and q_0 Along Length of Fractured Test Specimen F of Brass (as Received).

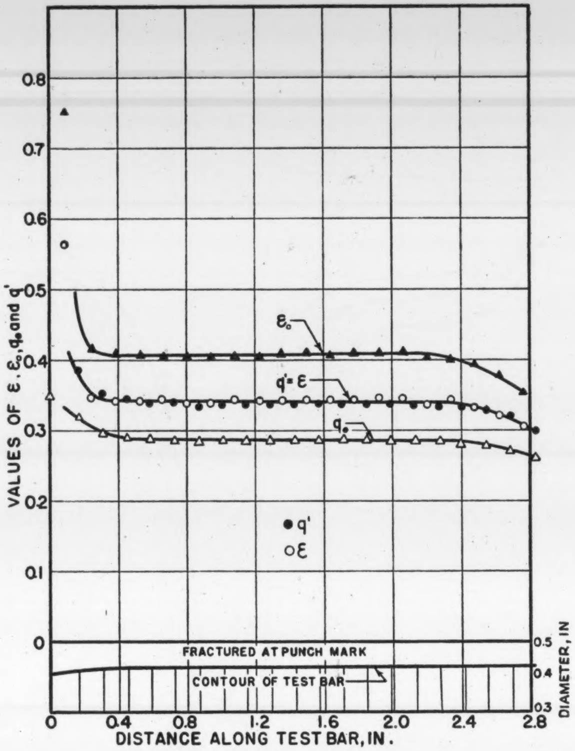

FIG. 8.—Values of ϵ_0, $\epsilon = q'$, and q_0 Along Annealed Brass Test Specimen E After Fracture.

formation becomes for cold-drawn as contrasted to annealed brass.

Another important feature which these curves bring out is that the heads of the test specimen in the standard A.S.T.M. 0.505-in. diameter test specimen are quite effective in restraining the deformation for some distance from the ends. Other tests were made on specimens of the same diameter but having a gage length three or more times the standard 2-in. length. In these cases, it was found for the material tested that a considerable length was obtained between the heads of the test specimen and the region of local constriction for which substantially a uniform diameter was maintained. It is then possible, for test specimens of this kind, to determine the uniform diameter which the specimen possessed at the maximum load from the final uniform diameter at fracture, with sufficient accuracy. Figures 3 to 9, while showing either inflection points in the curves, or in some cases very short horizontal portions (with the possible exception of the brass shown in Fig. 8), do not indicate any region of appreciable size over which the diameter is constant. The end-effect regions in most cases pass directly to the locally constricted areas without any uniform transition portion. In order to prevent any misunderstanding, however, let it be stated here that it is not the intention of this paper to propose any change in

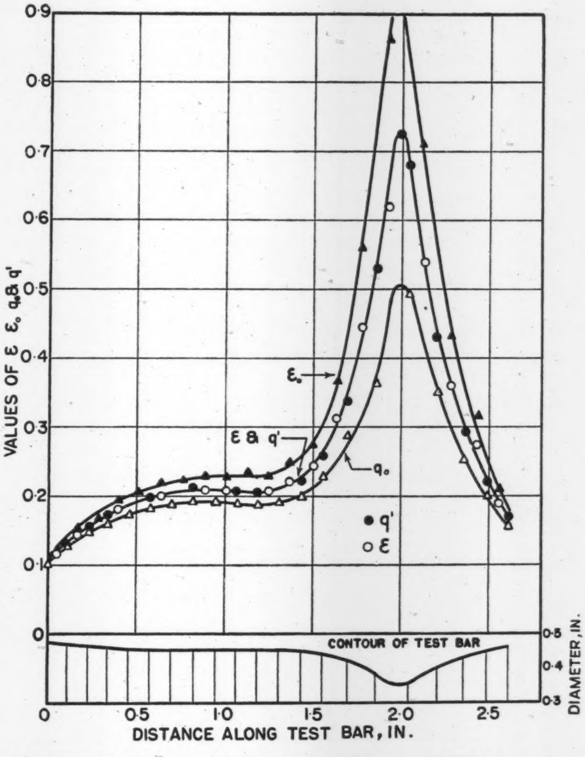

FIG. 9.—Strain and Reduction of Area Along Test Specimen of VD 95 per cent Carbon Steel (as Received) After Fracture.

the present standard test specimen. In the future, however, it may prove worthwhile to consider the possibility of also using an alternative test specimen with a larger gage length to diameter ratio such as is now the standard practice abroad.

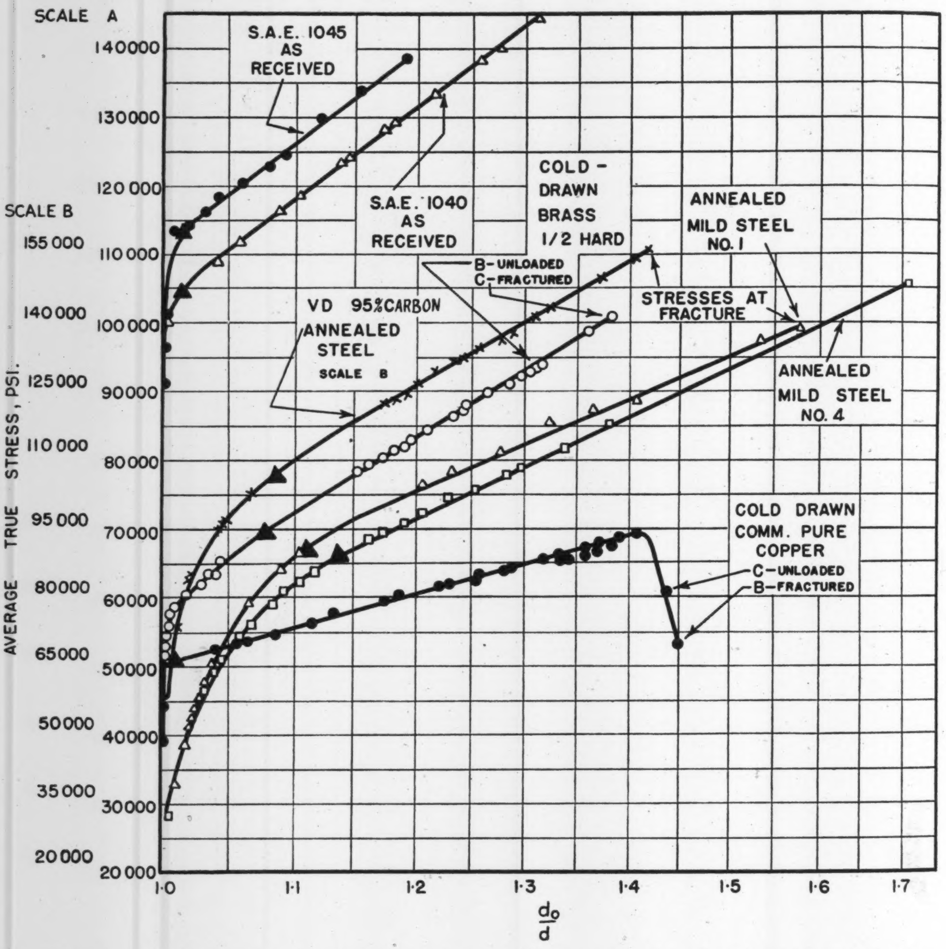

FIG. 10.—Average True Stress Plotted on Semi-Log Paper *versus* d_o/d for Tension Tests to Fracture of Various Materials.

TRUE STRESS-STRAIN CURVES

A discussion was made earlier of different types of stress-strain curves. It is proposed herein to describe one of these which has been suggested recently. This refers to plotting the average true stress $S = \frac{P}{A}$ as a function of the true strain or the true reduction of area $\epsilon = q' = \log \frac{A_o}{A}$, which expression becomes equal to $2 \log \frac{d_o}{d}$ for round bars. Since the true strain ϵ is equal to the true reduction of area q', only one of these quantities will be used in the description of the curves, that is, the true reduction of area q'. Such curves will then be referred to as S-q' curves.

Figure 10 shows curves for various metals, in which the average true stress $S = \frac{P}{A}$ is plotted on semi-logarithmic coordinates as a function of $\frac{d_o}{d}$. This is of course equivalent to plotting S against q'. The materials shown in these curves are different from those listed in Table II. It will be observed that each curve becomes a straight line from the value of the average true stress corresponding to the maximum load, indicated on Fig. 10 by the solid triangle, to fracture. There is only one exception to this: namely, the copper which remained straight until incipient fracture took place in the form of a crack inside the test bar. X-ray tests have shown that when the S-q' curve starts to drop off from the straight line, a crack has already formed inside of the test specimen.

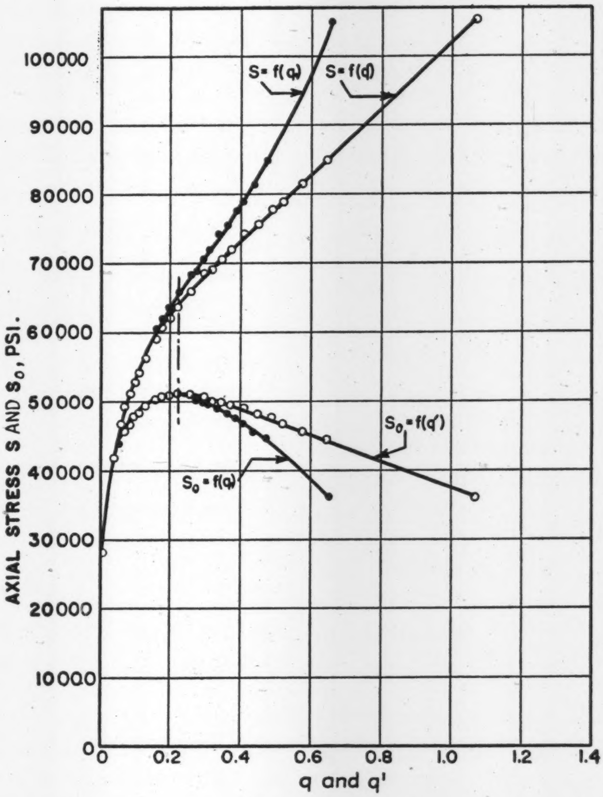

FIG. 11.—Stress-Reduction of Area Curves for 20C Annealed Mild Steel Bar No. 4.

As a further illustration four curves are shown in Fig. 11. The diagram in which $S = \frac{P}{A}$ is plotted for different values of q_o shows an upward curvature after the maximum load point is passed, this point being indicated by the vertical dashed line. The S-q' curve remains straight. These are then to be compared with curves in which the stress $S_o = \frac{P}{A_o}$ is plotted as a function of q_o and q'. Quite a number of other metals have been tested, and S-q_o and S-q' diagrams have been constructed for them. In each case it was found that the S-q_o curves showed an upward curvature after the maximum load point is passed, while the S-q' curves became straight lines in this region. This fact enables us to write a simple equation relating the average true stress at fracture S_b to the ordinary tensile strength $\frac{P_{\max}}{A_o}$ as follows:

$$S_b = \frac{P_{\max}}{A_o} \cdot e^{q'_u} + m(q'_b - q'_u) \ldots . (15)$$

In this equation, $m = \frac{\partial S}{\partial \epsilon} = \frac{\partial S}{\partial q'}$, or the true coefficient of strain-hardening for the material in the region of local necking. This coefficient of strain-hardening is the smallest value of $\frac{\partial S}{\partial \epsilon}$ exhibited by the material when subjected to a tension test.

One rather obvious advantage of plotting S-q' curves, aside from the fact that they represent physical relationships better, is that, since a straight line is obtained from the beginning of necking to fracture, only two points need to be observed to establish the curve in this region, thus cutting down the amount of work involved. The experimental technique necessary is also very simple in that a dial gage and clamp is the only instrument needed. Corresponding load and diameter readings can be taken quickly, and the results plotted either on semilogarithmic paper or on ordinary graph paper by picking q' values from a chart plotted as a function of $\frac{d_o}{d}$.

Another procedure which can be utilized in constructing S-q' curves from a tension test is the two-load method (3). According to this, the diameters along a properly tapered test specimen are determined at various positions before the test is made, and also at the same locations subsequent to fracture. Only the maximum and fracture loads are read from the scale on the testing machine during the test. Using the final areas of each position measured along the bar all the way up to the fractured cross-section, and dividing these areas into the maximum load, a curve of $\frac{P_{\max}}{A}$ can be plotted as a function of the corresponding q' measured. One other point needs to be located on the diagram, that is, the one determined by dividing the fracture load by the fracture area plotted with an abscissa equal to the q' at fracture. From this latter point a line is then drawn tangent to the $\frac{P_{\max}}{A}$ curve at its inflection point. It can be shown and checked experimentally that the curve of $\frac{P_{\max}}{A}$ *versus* q' represents the S-q' curve of the material up to the maximum load point, and the straight line drawn tangent to this curve from the point representing the average true stress at fracture provides the remaining part of the S-q' curve from the maximum load point to final rupture. The method is complete in that it determines the S-q' curve from initial yielding to fracture. It is hoped that the procedure will prove useful where it is desired to determine a true stress-strain curve for the material and measurements of diameter during the test are neither convenient nor feasible.

In concluding, it may be of interest to note the general effect of notches on the form of the S-q' curve. A series of test specimens containing notches of various sorts were machined from a different bar of annealed mild steel as shown in Fig. 12. Four of these test specimens were the same except that the length of the straight or uniform portion of the specimen was varied from $3\frac{1}{2}$ in. to zero (as in the case of the notch of $\frac{1}{8}$-in. radius). The remaining two contained a V notch and a square notch with a sharp transition. Figure 13 shows the S-q' curves obtained in each case. It will be observed that the tendency is for the S-q' curves to shift to the left as the effect of the notch becomes more pronounced. In all cases the maximum load point, indicated by the letter M, remained at approximately the same value of q'_u, but the average true stress corresponding to the maximum load increased with the more localized notch effects. One apparent result is then that, although the average true stresses indicated by points M increased considerably with the notch effect, the average true breaking stress or fracture stress remained practically the same. This shows again that the average true stress at fracture is more of a material constant than the value at the maximum load. The notch effect also reduced the straight portion of the S-q' curve until it was practically zero for the V notch. This would be expected since the adjacent material prevents local contraction. The V notch considered here is not as sharp nor as deep as the ones used by Kuntze, and it is not the intention to compare it with his results.

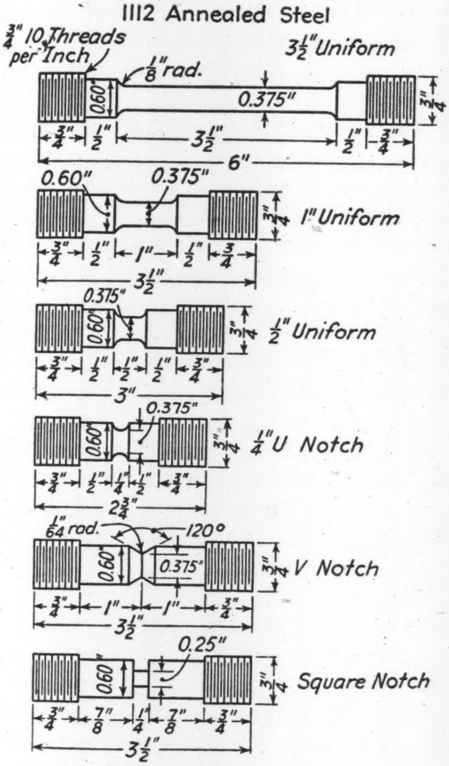

Fig. 12.—Tension Test Bars Used to Show the Effect of Notches on S-q' Curves.

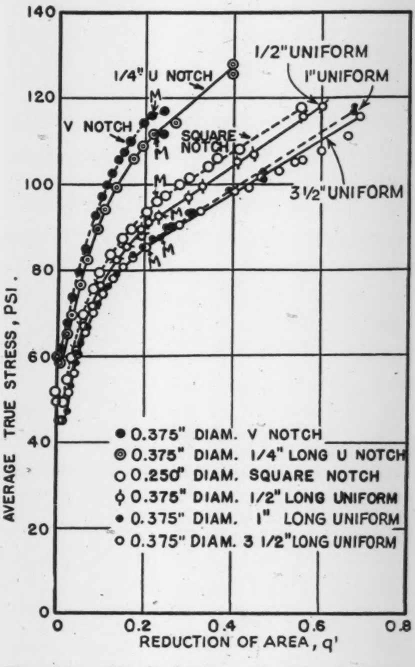

Fig. 13.—The S-q' Curves for Notched Specimens of S.A.E. 1112 Steel Machined as Shown in Fig. 12.

SIGNIFICANCE OF THE TENSION TEST

The significance of the various quantities determined in the tension test depends somewhat upon whether they are considered from the design, control, metal forming, or research points of view. From the design aspect, engineers have come to consider the yield strength for ductile metals and the ordinary tensile strength for brittle metals as convenient stress values upon which to base the design of statically stressed structures. The percentage elongation and reduction of area figures do not generally enter into design calculations but merely serve as indications of the ability of the material to absorb shock loads, or iron out stress-raisers through local plastic deformation. The true stress and strain values discussed earlier would have little use here. Attempts have been made, with only partial success, to relate the ordinary tensile strength and the yield strength of ductile metals to failure under fatigue conditions.

In the control of materials, most of the values determined in the tension test have significance although, in themselves, they are not sufficient in order to be sure that the material is suitable. Tension test figures are usually supplemented by impact values etc. It is felt that the average of the true breaking stresses and the newer definitions of ductility suggested earlier might help in providing more information on this problem.

A knowledge of true stress and strain values is particularly important in all metal-forming applications. The plastic portion of the S-q' curve taken in a tension test should provide useful information both in selecting metals for plastic operations of this kind and in relating stress, strain and work-hardening features.

In research work in the general field of the testing of materials, true stress and strain values are also of vital importance. By making use of the physical definitions of stress and strain stated earlier, it is possible to correlate tension values in a better fashion with those obtained in other tests such as combined stress tests, torsion tests, bending tests, and even in tension impact problems. No very clear correlation is possible if fictitious stress and strain values are used. The physical stress and strain values should also serve to throw more light on the mechanism of failure in polycrystalline metals.

In the light of the above remarks, it would appear that the tension test has possibly not been exploited to the fullest extent and that, for many purposes, certain information that should prove of value in the interpretation of results is ordinarily omitted from the data. It is realized that the tension test is limited in its scope, and that, without doubt, no rational correlation would be found in many cases where conditions are widely different from those of the test. Still, it would seem desirable to endeavor to obtain the maximum information which the tension test will yield without undue experimental effort.

It would also appear that there exists a need at the present time for more information surrounding the application of our materials where an accurate knowledge of true tension test values is available. Only further research will enable us to evaluate the importance of such knowledge. It would therefore possibly seem somewhat early to seriously delimit the significance of the tension test.

SUMMARY

A brief review is made of certain important work carried out in the past relating to the tension test. A discussion is included of various definitions of strain and reduction of area which have

been suggested, and these are related to the true values of strain and reduction of area. Approximate expressions are deduced for the true values of strain and reduction of area and these compared with the accurate relations. Ductility values are investigated and two new definitions of tensile ductility are suggested, namely, the true uniform strain and the true local necking strain. Experiments are described in which it is shown that the latter definitions of ductility yield more information on the deformation characteristics of the material than those customarily reported, which are the percentage elongation in 2 in. and the ordinary reduction of area. Certain advantages suggested by this method are described. Experimental results are included in the form of curves which show the relative values of the various magnitudes of strain and reduction of area along the length of fractured test specimens of various materials. The equality of true strain and true reduction of area is shown.

The true stress-strain curves are discussed in which the average true stress is plotted as a function of the true strain or true reduction of area. An analytical relationship between the true stress at the maximum load and the average true breaking stress is given. The two-load method of constructing these curves is described. Experimental results are included to show the effect of notches on the true-stress-strain curves. The physical significance of the tension test is discussed.

Acknowledgment:

The experimental work described in the paper was performed in the laboratories of the Department of Mechanical Engineering at the Massachusetts Institute of Technology and the author wishes to acknowledge the interest and support given to the work by Dr. Compton and Dean Moreland of the administration, and by Dr. Hunsaker, Head of the Mechanical Engineering Dept. He wishes to also thank J. M. Lessells and the other members of the Symposium Committee for their suggestions and comments, and W. Boyd and P. R. Shepler for their assistance in the various tests.

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(58) D. J. McAdam, Jr., and R. W. Clyne, "The Theory of Impact Testing," etc. Symposium on Impact Testing, *Ibid..,* p. 112.

General:

(59) R. L. Templin, *loc. cit.*

(60) J. D. Jevons, *loc. cit.*

(61) D. J. McAdam, Jr. and R. W. Clyne, *loc. cit.*

(62) G. Sachs, "Spanlose Formung der Metalle," *Mitteilungen* der deutschen Materialprüfungsanstalten, Sonderheft 16, p. 11 (1931).

APPENDIX

SYMBOLS USED

S_o, S = Tensile stresses where load is divided by original and actual areas, respectively.

S_b = Average true breaking stress.

P, $P_{\max}$ = Tensile loads.

L_o, L = Original and final lengths.

d_o, d = Original and final diameters.

A_o, A = Original and instantaneous areas, respectively.

A_u, A_b = Areas at the maximum and fracture loads, respectively.

e = Base of natural logarithms.

$m = \frac{\partial S}{\partial \epsilon} = \frac{\partial S}{\partial q'}$ = Coefficient of strain hardening in the region of local necking.

ϵ, ϵ_o = True strain $\log \frac{L}{L_o}$ and ordinary strain $\frac{\Delta L_o}{L_o}$, respectively.

ϵ_u, ϵ_n = True uniform strain, and true additional local necking strain, respectively.

ϵ_b = True maximum strain at fracture (in the region of local necking). $\epsilon_b = \epsilon_u + \epsilon_n$.

q', q_o = True reduction of area $\log \frac{A_o}{A}$ and ordinary reduction of area $\frac{\Delta A_o}{A_o}$, respectively.

q'_u, q'_n = True uniform reduction of area, and true local reduction of area (additional).

q'_b = True maximum reduction of area at fracture. $q'_b = q'_u + q'_n$.

q_u, q_b = Ordinary reduction of area values at maximum and fracture loads, respectively.

q_1 = Reduction of area equal to $\frac{A_o - A}{A}$.

ϵ_1 = Strain equal to $\frac{L - L_o}{L}$.

[See Discussion on Significance of the Tension Test, p. 579.—Ed.]

THE STRENGTH FEATURES OF THE TENSION TEST

By F. B. Seely[1]

Synopsis

A brief discussion is given of the influence on strength values obtained from the tension test of the haphazardly progressive slips in the grains of polycrystalline aggregates. Importance is placed on the conditions that change the mode of structural failure from general yielding (plastic deformation accompanied by strain hardening), associated with shearing stress, to failure by brittle fracture in which the cohesive strength of the material is exceeded, associated with tensile stress, unaccompanied by plastic deformation and strain hardening. The relation of state of stress, rate of strain, strain hardening, and temperature to these two modes of failure is emphasized; and an outline of tests to bring out information on these topics is offered. It is pointed out that a test specimen is a model and that the significance of test results depends on the extent to which the structural action in the specimen simulates the structural action which limits the useful load-carrying capacity of the prototype or structural member. In harmony with these considerations, the general conditions under which the strength features of the tension test are especially important are defined, after which a discussion of the significance of yield strength, of upper and lower yield points, and of high yield-ratio steels is given.

The title "tension test" without qualifying notation will be understood to mean a test in which a prismatic straight bar of constant cross-section is subjected to gradually increasing pulls (so-called static loads) at its ends until the bar or specimen breaks; the specimen is tested at ordinary temperature, and the loads at its ends are approximately axial giving substantially a uniformly distributed unidirectional stress. The title, therefore, does not include an impact tension test, a repeated-load tension test, a tension test of a specimen at elevated temperature (in which creep is involved), or a tension test of a notched specimen (in which the state of stress may be three-dimensional or the stress distribution on the least area may be highly nonuniform).

No mention will be made here of the specifications for the tension specimen and of the procedure for carrying out the tension test. These items are given in the Society's standards. Furthermore, it will, of course, be understood that the strength properties of a material cannot be isolated and considered independently; although the strength features of the tension test are here given emphasis, their relation to other desirable properties is always involved in the interpretation of the test results.

It seems desirable to make the discussion apply mainly to metals, not because other materials are thought to be

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unimportant, but because the topic even when restricted to metals is still too large to be discussed adequately in the space available. It should be stated also that the main ideas here given emphasis in the discussion of the strength features of metals have been presented ably, in recent years, by Kuntze (**1**),[2] Thum and Bautz (**2**), Heindlhofer (**3**), McAdam and Clyne (**4**), Hoyt (**5**), Bridgman (**6**), and others, and, over a longer period, by a large number of investigators in the field of fatigue of metals.

For the purpose of creating a background for the interpretation of the strength features of metals as found from the tension test, it will be desirable to trace very briefly the main ideas that have led to the present status of the tension test. During the past several decades, great confidence was built up in the value and significance of the tension test. This confidence was based on successful experience and is entitled to genuine respect. But a familiar process in the development of any field of activity consists in extending the methods and procedures that proved satisfactory in the experience with one set of conditions to a new or different set of conditions without fully realizing the factors upon which satisfactory performance depends. In other words, the human mind usually over-simplifies experience and then forgets or ignores the factors that were omitted in the simplification. Perhaps this process has been operating too strongly with respect to the tension test.

In the earlier years of rapid engineering development, methods of testing and of calculation of stresses were dominated largely by the conditions met in the use of hot-rolled low-carbon (ductile) steel under so-called static loads, in structures such as buildings, bridges, ships, etc. For these conditions, the strength values obtained from the tension test seemed to go a long way toward giving satisfactory information for determining the suitability of the material and for selecting design stresses for use in the stress formulas commonly employed. Such structures and machines designed on the basis of tension test values seldom failed in normal service. Furthermore, the tension test was so simple to carry out and its meaning or significance seemed so self-evident, that there was always a temptation to overemphasize its significance. The generalization that, for resisting loads, a material with relatively high strength values in the tension test must, to that extent, be better—better for almost any use—than one with relatively low test strength values, was often made almost unconsciously.

Over-Simplified Picture of Structural Action:

While confidence in the tension test was being built up, there also developed the idea that material (at least wrought ferrous metal) was perfectly elastic up to a certain stress called the proportional (elastic) limit. This stress was considered to be a definite inherent property of the material that could be determined accurately from the stress-strain diagram obtained from the tension test. Also, the belief was common that if the calculated stress in the material was less than this elastic limit, no damage in service—in any service—could possibly be done to the material. Moreover, calculated stresses obtained from the ordinary formulas in mechanics of materials, that is, formulas which consider only the maximum stress at a point (the most stressed fiber) without taking into account the effect of understressed surrounding material, were considered to give accurate measures of the significant stresses in the member.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 550.

As this over-simplified picture of structural action and of stresses was taking shape, the important part played by the material in undergoing innumerable minute plastic adjustments and accommodations that made the picture appear to be in harmony with experience—experience connected with structures made of ductile steel and subjected to static loads and relatively simple stress systems at ordinary temperatures—was almost wholly overlooked. However, when relatively ductile steel members, such as car axles, connecting rods, and especially members containing abrupt changes in section, broke in two in service under the action of many repetitions of stress, exhibiting a brittle type of fracture (a fracture unaccompanied by any evidence of yielding of the member) even though the maximum calculated stress in the member was less than the "elastic limit" of the material, a radical change had to be made in the mental picture of structural action taking place in metal under load, and also of calculated stresses. Likewise, radical changes had to be made in the interpretation of the strength features of the tension test.

At first a plausible explanation was offered for the discrepancy between the accepted picture of structural action as interpreted from the tension test and the observed fatigue phenomena—an explanation that demanded a minimum readjustment in the accepted picture—namely, that the material while being stressed changed from a fibrous (ductile and strong) material to a crystalline (brittle and weak) material. This wholly erroneous explanation still persists, particularly among mechanics, and it is difficult to displace because it seems to fit so well with the general appearance of many fractured members that have failed in fatigue.

Repeated loading, however, is only one of a considerable number of service conditions under which the maximum useful strength of a member cannot be determined from the strength values obtained from the simple tension test. These conditions consist of various combinations of the following: type of material, including nature of internal defects, especially in the surface layer of material; shape of member; temperature; and nature of loading. The significance of these conditions will be considered later in this paper.

A More Satisfactory Picture of Structural Action:

In response to the need for a better understanding of the useful strength of members under various conditions of use, there has been slowly evolving a more satisfactory picture of structural action in metals, and of the significance of calculated stresses.

Perhaps the greatest impetus toward creating this more satisfactory understanding of structural action in metals was contributed through the introduction of the metallurgical microscope (and later the use of X-rays) in the study of materials. When a metal was visualized, with the help of the microscope, as an aggregate of crystals or grains having irregular boundaries and containing nonmetallic inclusions, having the shearing strength of each crystal varying with the direction of the plane through the crystal, and having the planes of weakness in the crystals in the aggregate oriented at random, it became difficult to maintain the concept of perfect elasticity of the metal and of a definite value of stress corresponding to a sharp line of demarcation between damage and complete freedom from damage to a member.

The implications of this changed picture of the structural action in polycrystalline metals in relation to stress and strain have slowly been incorporated in the interpretation of the strength values of the tension test. Valuable contributions to the interpretation of the tension test have been presented before this Society and elsewhere by various members including Lessells (7), Templin (8), Moore (9), Rawdon (10), and others, and by several committees of the Society.

Two important implications growing out of the more detailed knowledge of the structural action in a metal member, such as a specimen in a tension test, made up of an aggregate of grains or crystalline units should here be mentioned. First, much of the useful static elastic strength of such a member as a whole could not be attained if inelastic actions in crystalline units at many locations in the member did not occur; or, to put the idea more tersely and perhaps more dramatically, the member, under conditions of static loads and ordinary temperatures, may have a relatively high structurally useful elastic strength because some of its weaker or poorly oriented elements act inelastically. The inelastic action in any grain or in a relatively small group of grains usually takes place through the mechanism of slip across planes of weakness depending on the orientation of the grains. The whole aggregate of grains (member) acts elastically as a group even though a considerable number of grains (small, however, compared to the total number of grains involved) takes on plastic deformation, often shown externally in the form of flow layers or Lüder's lines. This idea is sometimes expressed by stating that the aggregate of grains acts statistically as a homogeneous elastic body even though certain small groups of grains exhibit a very different behavior from the statistical or mass action of the aggregate. The so-called static elastic strength (such as the A.S.T.M. yield strength) as found from the tension test is, therefore, not a true elastic constant of the material or specimen, but is a stress accompanied by an arbitrarily specified permissible plastic deformation, the so-called proportional limit being the maximum stress for which the accompanying measured plastic deformation is zero, that is, less than the least (arbitrary) reading that is given by the extensometer used in the test.

The second implication is that, under conditions of use in which local actions in the units of the aggregate may be the damaging action, as in failure by fatigue and perhaps by high velocity impact loading, especially of members containing common surface defects and notches, the statistical properties as obtained from the tension test may have little or no significance. Whatever significance (if any) the tension test values may have for such conditions is probably to be found in the deviations from mass or statistical action. Some indication of these deviations is perhaps revealed by the characteristics of the stress-strain curve between the "proportional limit" and the yield strength, between the upper and lower yield point, and between the ultimate load and breaking load, particularly during the final stages of necking down. But as yet these matters have received little attention.

Two Basic Modes of Failure:

To this very brief survey of the main ideas that have gradually been changing our judgment of the value and significance of the tension test must now be added a somewhat more recent view of the subject which gives promise of making valuable contributions to the interpretation of mechanical tests of materials. Thus in considering the tension test and the conditions that make the tension test results significant, two radically different modes of failure of metal members must be recognized; the term failure as here used means any condition that renders a member in a structure or machine unfit for its service of resisting loads.

One mode of failure is by general yielding or plastic flow involving the statistical action as discussed above; this failure is denoted as a shear failure. The yielding that leads to failure is the result of slipping across planes of a very large number of crystalline grains. The useful resistance or strength of the material is associated with shearing stress, since the slipping across the grains is accompanied by shearing stress, and although the first slip of some grains may not be in the direction of the maximum shearing stress in the specimen, the (large) yielding, which constitutes the failure, is controlled mainly by the maximum shearing stress in the specimen as a whole; thus the resistance of a member to this mode of failure (the resistance to plastic deformation) is measured by the shearing yield strength of the member.

The other mode of failure consists of the breakdown of the cohesive resistance of the material, resulting in a fracture unaccompanied by yielding (brittle fracture), and the useful strength of the material is associated with the tensile breaking strength of the material under conditions that prevent yielding. This type of failure arises when the tensile stress in the material exceeds the cohesive strength of the material. Some materials, such as brick and stone, almost always fail this way and hence are said to be inherently brittle. Metals, however, especially ductile metals, seem to fail by having their cohesive strength exceeded (without previous plastic deformation) only where the stress is highly localized and particularly where the state of stress approaches a triaxial tension. Unfortunately the cohesive strength of a ductile metal cannot be determined from any simple test that has so far been devised; Kuntze (11) has proposed a method of extrapolation of the test data obtained from static tension tests of notched specimens in which the notches vary in depth and angle.

The cohesive strength here referred to is associated with the material in mass or in bulk as it occurs in engineering members, rather than with the forces of attraction between the atoms. Some authors speak of it as the "technical" cohesive strength. Further, it is not measured by the breaking stress in a tension test (breaking load divided by the area of fracture), since the breaking stress occurs only after the material has been subjected to much plastic yielding.

There are many conditions that can change the relation between these two strengths (resistance to plastic deformation and cohesive strength) for any one metal, as for example, the temperature, state and distribution of stress, rate of straining, and amount and rate of strain hardening. And if, in a given service, it is not fairly definitely known which type of failure will occur, an intelligent interpretation of test results from any test is very difficult, if not impossible, to make.

On the other hand, as Hoyt (5) points out in discussing the notched-bar impact test, "If one knew the controlling properties, cohesive strength and resistance to deformation, and if he thoroughly understood the laws of the notch effect, he would be able to predict by calculation whether a given steel would deform when notched; or open up a crack."

If it is true that the useful strength of a member is limited either by its cohesive strength or by its resistance to plastic deformation, an understanding of the conditions under which each of these strengths is likely to reach its limiting value is highly important; likewise it is important to determine what information concerning these two strengths can be obtained from the simple tension test.

Conditions Under Which Tensile Strength Values are Significant:

The main conditions that must be satisfied in order that yielding resistance (resistance to plastic deformation) shall be less than the cohesive resistance of the material are: (*1*) A material must be ductile, that is, one which will yield plastically under stress if shearing stress is permitted to be developed; most structural metals satisfy this requirement. (*2*) Loads must not be continuously repeated, nor applied with a velocity above a critical value which depends on the material, temperature, etc. (*3*) The state of stress that exists must be sustantially unidirectional or at least be well-removed from triaxial tension, so that shearing stress is free to develop. (*4*) The stress distribution that exists must be one in which the stresses are not only unidirectional but also one that is free from high stress peaks. Such stress peaks may occur at abrupt changes in section, or at nonhomogeneities in metal structures, and may exceed the cohesive strength of the material before local yielding occurs, the restraint to yielding being offered by the surrounding understressed material; or if local plastic yielding does occur at the points of stress concentration, the local state of stress set up at the end of slip planes probably is three-dimensional tension in which shearing stress is inhibited, and hence the tensile stress is likely to exceed the cohesive strength of the material, thereby causing failure by a local fracture (crack), especially under repeated and impact loads. This local action probably simulates the formation of the crack that sometimes occurs at the central portion of the necked-down section of a tension specimen before the outside portion fails in shear.

It will be observed that the four conditions listed in the preceding paragraph are the conditions that are satisfied (approximately) in the use of structural steel for buildings, bridges, and many other similar structures; and hence, as already pointed out, the strength values determined from the tension test, in which failure is by general yielding, were for such service found to be significant. It should be noted that in this discussion failure by elastic buckling is not considered.

If, however, the member in service would fail by the breakdown of the mass cohesive strength of the material resulting in fracture without an accompanying plastic yielding, by virtue of a state of stress that prevents shearing stress from developing or by virtue of a cohesion weakness in the inherent structural constitution of the material, the usual strength values (such as yield strength and even tensile strength) obtained from the tension test would have little significance. Schuster (12) emphasizes this fact when he says, "the author wishes to say, however, that fracture in service of the varied class of parts which he has examined during an extended period, short of other defect in the material, has never been due to an unsatisfactory yield."

In the specimen of a simple tension test, the tensile stress is twice the shearing stress, and hence, if the specimen yields, all that the test tells about the cohesive strength (of the undeformed metal) is that it is greater than twice the resistance to deformation of the metal and that its value is greater than the tensile yield point—how much greater than the tensile yield point is not revealed by the test. Therefore, if this same material were used in a member containing notches where a stress peak may be produced, and in the neighborhood of which the state of stress may approximate a state of triaxial tension so that the tensile stress developed is high and the shearing stress is low, the tensile stress might exceed the cohesive strength of the material before the shearing stress could reach a value equal to the maximum resistance to deformation. The results of the simple tension test, therefore, would, under these conditions, have little significance, and might be very misleading. To what extent sharp notches create stress peaks and polyaxial tension is not definitely known; in other words, the mechanics of stresses at notches is to a large extent an unexplored field of knowledge, but since so-called ductile metals often fail by cracking at notches, there is evidence to support the idea that the state of stress may change the mode of failure from (ductile) yielding to (brittle) fracture.

Further evidence of the effect of state of stress on the mode of failure is found in the fact that a material that gives a brittle fracture type of failure under a unidirectional stress may fail by yielding under a polyaxial state of stress in which the principal stresses are not of the same sign. Thus a metal like high carbon steel that shows a brittle fracture in the tension test, in which the shearing stress is equal to one-half the principal (tensile) stress, may deform considerably, and even fail by yielding, in a torsion test, in which one principal stress is tension and the other compression giving rise to a shearing stress equal to each of the principal stresses. It might be well to give one other illustration of the influence of the state of stress on the mode of failure, since this idea, although fairly well known, is frequently overlooked. Even under repeated stress, unnotched specimens of ductile steel will fail initially by yielding (especially under repeated torsional loads) if the stress is not completely reversed but is such that the maximum stress in the range of stress is rather high; whereas, under the same type of loading, specimens of the same material containing a small transverse hole will exhibit a brittle fracture (fatigue) type of failure for all ranges of stress (13). Apparently, the high stress concentration at the edge of the hole changes the type of failure from (ductile) yielding to (brittle) fracture, even though the state of stress (principal stresses of opposite sign) a little back from the edge of the hole is favorable to yielding.

Under what conditions, then, are the strength values obtained from the tension test significant? If in service the metal would fail by yielding of a considerable portion of the member (involving slipping across planes of many crystalline grains accompanied by strain hardening), the A.S.T.M. yield strength or yield point (or some one of the other several measures of so-called elastic strength) as obtained from a tension test will have real significance in design and in determining the suitability of the material for the given service. Likewise, the ultimate strength will have significance, particularly in indicating reserve strength (as accident insurance); in addition, of course, the ultimate strength of a brittle material may be of primary importance.

Limitations on Significance of Tension Test:

Attempts are frequently made, however, to use the results of the tension test to determine the suitability of a ductile metal that does not fail in service by general yielding. For example, it is true that test results show a usable correlation between the tensile strength of the standard tension specimen and the endurance limit of unnotched polished fatigue specimens subjected to completely reversed stress, for one class of plain carbon steel: namely, relatively low-carbon hot-worked steel. But there seems to be no sound basic reason to explain such a correlation; the tensile strength is a stress associated, in this class of steel, with a large amount of general yielding, whereas the endurance limit is associated with fracture unaccompanied by general yielding. For high-carbon steel where a correlation might possibly be expected, a poor correlation exists, and even for low-alloy, low-carbon steel a satisfactory correlation is not found.

The attempt to use test results associated with one type of failure for the basis of design of members that would fail in service by a different type of structural action is analogous to the use of the Euler slender column (buckling) formula for the design of a stocky column that would fail by yielding long before the Euler buckling load could be applied. One engineer, in discussing a problem in which he had used this method, recently said to the author that by dividing the Euler buckling load by a factor of safety which gave a working unit-load well below the yield point of the material, he obtained a satisfactory design value; he seemed to fail to realize that the significant fact in his statement was that the value was satisfactory because it was less than the yield point and not because it was some proportion of the buckling (Euler) load.

It seems evident, therefore, that the tension test, though important, has limited significance in indicating suitability of a material for resisting loads—limited to conditions in service in which the structural actions that lead to failure are substantially the same as those that take place in the test specimen. The value of the test in indicating the care and thoroughness with which the processing of a material has been carried out is not here under consideration.

Need for Tests That Have Structural Significance:

In the past the suitability of a material for a given use has in general been determined from the record of performance of the material in actual service rather than from the properties found from tests. The service record, perhaps, will always be the final test of the suitability of a material. This fact, however, should not be considered, by materials engineers, as proof that properties of materials obtained from tests cannot be made to go a long way toward predicting satisfactory service.

The late J. A. Capp, who was one of the first members of this Society to see the need for determining the significance of the properties of materials, in contrast to merely making standardized tests, gave expression to the general problem with which this symposium is concerned when, in his presidential address (14) in 1920, he said: "We know the conditions to be met in service by any structural part and we know many of the useful properties of materials. Must we not now address ourselves to the solution of the problem, 'on what properties does the suitability of a material for particular applications depend and how may these properties be used to measure suitability'?"

The test results from the tension test will be significant (though not sufficient in themselves) in measuring suitability of a material for a given service if the structural action in the test specimen brings into play (perhaps exaggerated somewhat, but not too much) the structural action involved in the process of failure of the actual member used in the structure or machine. If this similitude of actions in the tension specimen and the actual structural member is lacking, as is frequently the case, the most that can be said for the test results is that they indicate whether a new batch or shipment of a material gives test values approximately the same as those found for similar (supposedly identical) material that has given satisfactory service. There is no desire here to minimize the importance of this function of the tension test, but this function should be clearly differentiated from that of determining properties for predicting the suitability of a material for a given service, and for use in design.

The problem of devising tests to determine the suitability of a material for a given service may never be solved with complete satisfaction, but progress toward a solution depends largely on making the right approach. As intimated in the preceding paragraph, a test specimen may be thought of as a model; and, if the laws of similitude are observed, the results found from the model test may be used to predict the behavior of the actual member or prototype. But in almost all model tests complete similitude is impossible; and hence, it becomes necessary to decide on what group of factors the dominant or significant action depends, and to incorporate, if possible, this particular group in the model test.

In the field of properties of materials, however, this procedure for model testing usually requires a more detailed knowledge of the phenomena of failure of materials under various conditions of use than is now available, although progress in obtaining the necessary knowledge is being made. The use of the principles of similitude in tests of models is discussed briefly here for the purpose of emphasizing the basic needs in the problem of determining properties of materials for use in predicting suitability of the material in specified conditions of service. A mere list of some of the more dominant factors that influence the significant structural action involved in the failure of material under various service conditions indicates the difficulty of satisfying the requirements of similitude in a model test, and also serves to indicate the limitation of the simple tension test. In stating the following list of some of the more important factors, in addition to the material itself, that influence the significance of test values, it is recognized that there will be some overlapping of conditions:

1. Size and geometric proportions of specimen or model, including surface finish, particularly when failure is by fatigue.
2. Temperature—particularly when failure is by creep at elevated temperatures, or when the failure under high-speed impact loading may change, when the temperature becomes relatively low, from a ductile type (accompanied by high energy to rupture) to a brittle type (low energy) of fracture.
3. Stress distribution, involving the relative proportion (volume) of a member subjected to high stress—including the effect of stress concentration, and of residual stresses.
4. Number of structural units (crystalline grains) taking part in the structural action. This involves the size of grains in addition to the stress distribution.
5. State of stress at any point. A unidirectional stress might give rise to a yielding (shear) type of failure; whereas a three-dimensional state of tensile stress might result in a brittle fracture type of failure.

6. Orientation of grains; nature, size, and location of nonmetallic inclusions; and nature of surface discontinuities such as laps, cracks, and decarburization effects—particularly when local actions lead to failure (rather than statistical or bulk action) as in failure by fatigue, and by high velocity impact.

TABLE I.—OUTLINE OF TESTS OF A MATERIAL TO SHOW EFFECT, ON CHANGING THE MODE OF FAILURE FROM PLASTIC YIELDING TO BRITTLE FRACTURE, OF PREVIOUS PLASTIC DEFORMATION, RATE OF DEFORMATION, STATE OF STRESS, AND TEMPERATURE.

Amount and Direction of Previous Plastic Deformation	
	(*1*) Zero plastic deformation. (*2*) Specimen given a specified tensile plastic deformation in longitudinal direction and then tested with principal stress in direction of plastic deformation. (*3*) Same as (*2*) except tested with principal stress perpendicular to direction of plastic deformation.

Rate of Deformation in Test	Degree of three-dimensional tensile stress; indicated by ratio of maximum tensile stress to maximum shearing stress, $\frac{\sigma}{\tau}$			Temperature			
				Low	Medium	Higher	High
Very slow. (Ordinary testing machines with load gradually applied)	Torsion Test		$\frac{\sigma}{\tau} = 1$. (Very small) Unnotched specimen				
	Tension Test	(*a*)	$\frac{\sigma}{\tau} = 2$. (Small) Unnotched specimen				
		(*b*)	$\frac{\sigma}{\tau} =$ medium. Shallow notched specimen with root of notch not too sharp. $\sigma =$ large				
		(*c*)	$\frac{\sigma}{\tau} =$ large. Deep, sharp notch. $\tau =$ very small				
Medium. (Charpy tension impact)	Same as (*a*) above						
	Same as (*b*) above						
	Same as (*c*) above						
Relatively high. (Charpy or Izod bending impact test)	$\frac{\sigma}{\tau} \gtreqless 2$. Usual (single) width *un*notched specimen (for less ductile material)						
	$\frac{\sigma}{\tau} =$ medium. Single-width notched specimen						
	$\frac{\sigma}{\tau} =$ larger. Double or triple-width notched specimen						

7. Time permitted for deformation to occur—rate of straining, especially in connection with stress concentration and energy (impact) loading of soft metals.

8. Manner of loading and straining (static, impact, or repeated; and axial, bending, or torsion).

9. Chemical composition and stability, especially of age-hardening metals and of steel at elevated temperatures.

10. Amount of plastic strain or of strain hardening in the member as a whole and also locally at notches.

All of these conditions influence the relative values of the cohesive resistance and the resistance to deformation of a material. It would appear, therefore, that progress in determining strength (or other) properties of material for predicting suitability of metals for given conditions of service and for use in selecting design stresses, requires that tests be devised that will bring into play those properties or groups of properties that are involved in the structural action that leads to failure under service conditions. The starting point in this problem, therefore, is the study of the phenomena of failure under various service conditions. The problem, however, of determining the significant structural actions involved in various types of failures and of devising tests to measure the corresponding significant properties of materials is not only a difficult problem but also a costly one; it is, nevertheless, a problem whose solution is urgently needed and a problem whose solution constitutes one of the main objectives of this Society.

A Suggested Outline of Tests:

As one approach to the problem, the outline of tests shown in Table I is offered merely as a suggestion for getting before the eye a summary of the facts needed for determining the suitability of a material for use in a metal member under certain general service conditions. The outline does not include tests involving all service conditions; it does not, for example, involve failure by creep (long-time static loading at a temperature above the recrystallization temperature) or by fatigue (long-time repeated loading at ordinary temperature); likewise the effect of aging after previous plastic deformation is not considered. It includes only conditions that can be investigated in relatively short-time tests with testing machines and equipment that are now readily available. The outline is offered mainly to emphasize the main factors (in addition to repetition of loading and elevated temperatures) which can influence the mode of failure of a member and which, therefore, must be given weight in determining the suitability of a material for use in members for resisting loads as used in various service conditions. Under each set of conditions the mode of failure would be observed and, in certain tests, numerical values of strength and of ductility would also be determined. The values of the temperatures designated as low, medium, high, etc., will, of course, depend on the service conditions. For service at so-called ordinary (atmospheric) temperatures, "low" might be −60 F. and "high" might be 150 F. If information such as that suggested by this outline of tests regarding the mode of failure under various service conditions is available, the significance of the strength values of the tension test, and of other mechanical tests, becomes much easier to determine.

It is realized that the picture of the subject as presented in this paper has been painted with a broad brush. This plan, however, has been followed purposely in order to direct attention to the underlying structural actions on which the significance of the strength features of the tension test depend. It seems desirable, nevertheless, to discuss briefly three special topics associated with the tension test: namely, the significance of yield strength, of upper and lower yield points and of high yield-ratio steels.

Yield Strength.—If a metal member is used for resisting loads under conditions that would cause the member to fail by general yielding as interpreted in preceding portions of this paper, the question arises as to what stress obtained from the tension test should be selected as a measure of the maximum utilizable tensile strength of the metal, especially for a metal that does not have a well-defined yield point. The Society's standards propose for this purpose the yield strength defined as: "The stress at which a material exhibits a specified limiting permanent set." The method of determining this stress is designated as the offset method and consists of drawing on a stress-strain diagram a line parallel to the low-stress (straight line) part of the diagram at a distance therefrom, parallel to the strain axis, equal to the specified offset. The stress corresponding to the point where the offset line intersects the stress-strain curve is the yield strength.

There have been two rather different reactions to the use of the yield strength. One type of reaction comes from engineers who are concerned mainly with tests of materials for the purpose of determining whether the material passes the specifications. The feeling among some members of this group is that a value should be determined from the test that is less expensive to obtain than is the yield strength; one that will slow up production less and that will require simpler instruments (perhaps dividers instead of an extensometer), and one that requires less manipulation of test data (no stress-strain diagram). If a test value can be found from the tension test that will satisfy these requirements, and at the same time will be trustworthy and have real significance as a measure of structural damage, all people involved in the production and use of the material would probably be satisfied; however, such a test value does not seem to have been found, at least not for general use.

The second type of response comes from engineers who are concerned with the problem of determining the suitability and the properties of the metal for use in load-resisting structures and machines. Some of the members of this group feel that the yield strength does not indicate the utilizable strength of the material (still assuming that failure in service would occur by general yielding) unless the offset used in determining the yield strength is very small—of the order of the smallest reproducible reading of the extensometer. This viewpoint probably is the result of the lingering belief in a "true" proportional (elastic) limit. The use of such small offsets may be useful in determining whether a heat treatment has been carried out satisfactorily (without introducing high internal stress, for example), but the utilizable strength of the material is rarely limited to permanent deformations represented by such small offsets. The yield strength is a value of stress that is accompanied by a sufficiently large deformation or offset to be substantially free from the influence of the technique of the individual who performs the test; at the same time it is a significant value—a property of the material associated with the structural damage which occurs in the member when used so that failure is by yielding; and moreover it is applicable to a wide range of alloys. Other test values can be found from the tension test that also satisfy these requirements, but perhaps none is found more simply than is yield strength. It is doubtful whether any single strength value can be found that will be free of the above discussed objections.

Doubt is sometimes expressed as to whether a metal that does not have a definite yield point can undergo sufficient plastic flow to relieve localized stresses and make other adjustments required in the successful functioning of many members in structures and machines. Thus a question is raised concerning the structural significance of the yield strength of such a material. Since the amount of plastic flow required for satisfactory functioning of a member of a given material as used in a machine or structure probably is never definitely known, various special tests of the material, such as those suggested in the outline given earlier in this paper, together with tests of large-sized structural members having a surface finish as manufactured or fabricated, and experience in the use of the material must furnish the answer to the question. In general, there seems to be little doubt that the yield strength does have real structural significance as a limiting design stress for static load conditions. In this connection it is well to recall that a material that exhibits a pronounced yield point under uniform distribution of stress as in a tension test may reveal little evidence of a yield point under a nonuniform stress distribution even though the stresses are unidirectional, as in a bending test, and hence the yield point phenomenon may be operative to a limited degree in many members even though the material may be said to have a well-defined yield point.

One more question should perhaps be raised. Since permanent yielding of metals is basically associated with shearing stress rather than with tensile stress, there seems to be good reason for asking why the tension test is retained as the basic test for the strength features of material that would fail by yielding. A discussion of this question is somewhat beyond the scope of this paper but it is well to recall that most structurally useful metals are useful largely by virtue of the fact that they have cohesive strengths (resistance to brittle separation) that are large relative to their shear strengths (resistance to deformation), and hence the tension test can be used to bring out the yield properties of the materials, which are structurally significant if the material is used under a stress condition that will permit yielding.

Upper and Lower Yield Point.—The question is sometimes raised as to whether the upper or the lower yield point of steel should be used as a measure of the maximum utilizable tensile strength of the material (assuming, of course, that the material is a grade of steel that really possesses a yield point; and assuming also, as before, that if the material fails in service, it does so by general yielding and not by elastic buckling or by brittle fracture as in fatigue, etc.).

In the usual method of testing for determining the yield point, an upper yield point is obtained, but not the highest value that could be obtained by observing special care in preparing the specimen and in carrying out the test. Under the existing specifications for the tension test the range between the upper and lower yield points is, as a rule, quite small, and the stresses corresponding to the drop of the beam or halt of the dial, and to the horizontal part of the diagram in an autographic stress-strain curve, are satisfactory values for the maximum utilizable strength of the material under the conditions of use here assumed, provided that the speed of testing and other details of the test procedure are given proper consideration in carrying out the test.

One of the reasons for giving only secondary importance to the phenomena of upper and lower yield point for the purpose here considered arises from the fact that the values of both yield points are affected appreciably by the type and characteristics (especially springiness) of the testing machine and by the technique of testing. Another reason is that with the various common types of nonuniform stress distributions in members, it is not clear what significance the phenomenon of upper and lower yield points exhibited in a specimen under uniform stress has on the useful functioning and resistance of the member, even though failure would be accompanied by general yielding. As pointed out in the earlier part of this paper, the significance of test results depends on how well the structural action in the test specimen simulates the structural action of the material in the member, which brings about failure of the member.

On the other hand, in certain processing operations, such as deep drawing, where localized yielding in the form of strain markings or stretcher strains may occur, a different significance probably should be attached to the phenomenon of upper and lower yield points.

High Yield Ratio Steel.—With the production, during the past few years, of a variety of low-alloy (hot-rolled), high yield point steels, a question that has often been raised in the use of the higher alloy (heat-treated) ductile steels has been given added emphasis. The question is whether full use, even under static load conditions, can be made of the higher yield point of these steels, particularly if the increase in yield point is accompanied by a narrower spread between the yield point and the ultimate strength as obtained from a tension test.

For resisting static loads, the spread between the yield point and the ultimate is frequently considered to be a measure of reserve strength that is available for two purposes. First, if the state of stress or stress distribution in the member is such that yielding of a considerable portion of the member may occur, the reserve strength is a measure of the resistance of the member to accidental or unforeseen overloads; and, second, if high localized stresses exist in the member and are not relieved by local yielding because yielding may be inhibited by a triaxial state of stress, the reserve strength of the material is assumed to indicate the ability of the material to resist the high stresses and thus to prevent fracture or cracking. However, this assumption seems to overlook the fact that the reserve strength indicated in the tension test involves stresses accompanied by large amounts of plastic deformation, whereas the property of the material needed under the conditions stated is the cohesive strength, that is, the stress that can be resisted when yielding is prevented and strain hardening does not occur.

Figure 1 shows the tensile stress-strain diagrams reported by Collins and Dolan (15) of two ductile, hot-rolled

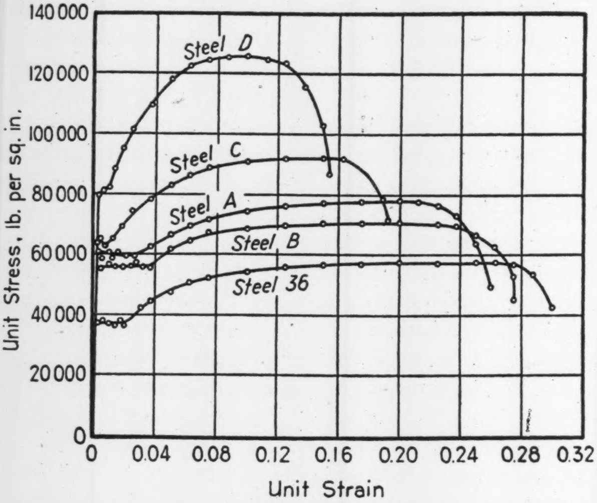

FIG. 1.—Typical Stress-Strain Diagrams of Tension Tests.
8-in. gage length, 0.5-in. diameter.
NOTE.—Steels A, B, C and D are low-alloy steels, and steel 36 is plain carbon structural steel.

low-alloy steels (designated as A and B) having a ratio of yield point to ultimate strength (called yield ratio) of 0.77 and 0.79, respectively, whereas the yield ratio for the ordinary carbon structural steel (designated as 36) is only 0.64. It will be noted that the ductility, as indicated by the elongation of a tension specimen at rupture and also at the yield point, is substantially the same as the ductility of the carbon structural steel. The following questions naturally arise: Do the lesser amounts of reserve strength of the two high yield ratio steels make them undesirable for use in members for resisting static loads? Is there any way of determining how much reserve strength is needed in structural and machine members whose useful strength is limited by general yielding? It is doubtful whether many engineers would wish to give dogmatic answers to these questions, particularly if various other tests indicate that the notch sensitivity of these steels is no greater than that of plain carbon structural steel.

If the tensile yield point of a plain carbon structural steel were found to be greater than 0.65 to 0.70 (say) of the ultimate strength, the steel probably would be considered to be unreliable, and the yield point probably would not be considered to be a satisfactory measure of the utilizable tensile strength of the material for the use here assumed. Even if this conclusion is correct with respect to plain carbon structural steel, the question may be raised as to whether one is justified in carrying over the conclusion to the low-alloy, high yield ratio structural steels. If a specification states that the yield ratio shall not exceed some limiting value (say 0.70), is this value specified because of the requirement for satisfactory design, for fabrication, for uniformly reliable material, or for some other requirement?

Perhaps tests alone will not furnish a satisfactory answer to such questions, but if more testing were done in accordance with the ideas discussed earlier in this paper, the significant characteristics and properties of materials would be better understood.

One fact should, perhaps, here be emphasized: namely, that if a high yield ratio steel is used to replace a member made of plain carbon steel and the working stress is based on the yield point, the relative decrease in the stiffness of the new member due to its reduced dimensions may cause a serious increase in the loads taken by other members in the machine or structure unless the whole assemblage of members is carefully considered from this point of view. Likewise, the deflection of the member made of the high yield ratio steel, rather than its strength, might limit its useful resistance.

In general, the fatigue strength (resistance to localized fracture, or cracking) of high yield ratio steel, does not increase proportionately with the yield point. This statement applies in a minor degree to some of the low-alloy (hot-rolled) steels, provided that stress concentrations are present, but it applies more particularly to the higher-alloy (heat-treated) ductile steels. Thus, an increase in resistance to deformation of these steels is not accompanied by a proportionate increase in their cohesive strength, that is, in their resistance to a brittle fracture or separation type of failure such as occurs as a spreading crack in a fatigue failure. It is evident therefore that in selecting design stresses for members of such material to be subjected to a combined steady and variable stress, it may not be possible to take full advantage of the higher yield point of the heat-treated high yield ratio steels, since failure by fracture (fatigue) may occur before the yield strength of the material is reached. Results show (13, 16) that this latter condition will occur mainly when stress concentrations are present and when the variable stress is relatively large compared to the steady stress.

No doubt more tests and experience are needed before the significance of a high yield ratio in steels can be determined satisfactorily, but all such tests and experience need to be interpreted in connection with the types of structural actions that limit the useful resistance of members, as discussed in the previous parts of this paper.

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[See Discussion on Significance of the Tension Test, p. 579.—ED.]

THE LIMITED SIGNIFICANCE OF THE DUCTILITY FEATURES OF THE TENSION TEST

By H. W. Gillett[1]

. In this discussion we shall confine ourselves to asking what ductility data mean. We will not waste time in recapitulating the technique of the determination of elongation and contraction, a matter thoroughly covered by A.S.T.M. specifications, in talking about the correction factors among elongations determined on various gage lengths or in worrying about the difficulty of getting contraction figures on sheet or other than round specimens. We may assume that if we wanted the data badly enough, they could be secured on any desired gage length or from any thickness of specimen so that all materials could be compared without the ductility data being affected by the method of testing. We may also assume that the temperature and speed of testing are chosen to be suitable for evaluation of ductility as related to the intended type of service. For brevity, the word "contraction" will be used instead of "reduction of area."

There is widespread belief—and as a qualitative first approximation, it is true—that large figures for percentage elongation and contraction in the tension test connote toughness and an ability to "take it" in fabrication and in service. We know that the strength figures of the tension test are quantitative for design. If we want to support a 25,000-lb. dead load by a bar of 1 sq. in. cross-section (if we do not care about elastic extension of the bar so that elastic moduli are not important) and if we are not concerned with creep, then, for this condition of shockless static loading, any material whatever with an elastic limit (determined, let us say, by the proof stress method) of 30,000 psi. is a substitute for any other. That is, the 30,000 figure means something irrespective of the material. It is only human nature to expect, or hope, that such figures as 15 per cent elongation in 2 in. and 35 per cent contraction, also mean something by and in themselves.

Generally speaking, tensile strength and tensile ductility in metals are inversely related; with very high strength we must be content with a low level of ductility and, with a high level of ductility, with low strength. Because ductility can be expressed in figures, there is a tendency to be impressed by the figures, to be favorably disposed toward getting the figures as high as possible, and to draw specifications upon arbitrary figures. Once having drawn such specifications we tend to think in terms of those figures rather than in terms of the plastic action concerned.

Ductility Is Sometimes Waived:

In spite of this predilection toward high ductility and large ability for plastic flow, the engineer sometimes manages to get along without any high degree of plasticity.

Since nature does not provide the

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same degree of attainable ductility on all our engineering materials, we adapt ourselves to the situation. The same set of tensile ductility figures may represent low ductility for a weak material and unattainably high ductility for a strong material. In a deep-drawing steel 15 and 35 per cent would not arouse much enthusiasm, but one does not refuse to use spring steel merely because it does not show 15 and 35 per cent. Nor are materials, such as cast iron and cast 8 per cent copper, 92 per cent aluminum alloy, refused engineering use at a close approach to 0,0. Zinc-base die castings find increasing use, even in such things as chain hoists. Yet individual specimens of the die-cast alloy XXI[2] are acceptable at 0.5 per cent elongation in 2 in. and a Charpy impact value of 4 ft-lb. (on an unnotched $\frac{1}{4}$ by $\frac{1}{4}$-in. bar). "Plastics" (molded organic condensation products) despite their name, are ordinarily to be classed among brittle materials. Plasticizers are added to increase formability at molding temperature, but at room temperature the plastics are not very ductile. Data cited by Penning and Meyer **(1)**[3] for cellulose acetate and by Bartoe **(2)** for the methacrylate resins show this. At the temperature of a fall day, such resins may give from a few per cent to nearly zero tensile ductility and very low impact figures. Indeed, "cold flow" in plastics is a failing carefully avoided, plasticity at the temperature of use is held to a minimum. Yet the plastics are widely used, even in stress-bearing applications.

The point is that we use cast iron, cast aluminum alloys, zinc-base die castings and molded plastics as engineering materials, in spite of low tensile ductility, and sometimes we use them in applications where some degree of accomodation or "give" to equalize stress distribution is needed, and where we would not use glass. These materials, returned as "brittle" by the criteria of tensile ductility and impact tests, have something that differentiates them from a glass-brittle material and renders them acceptable for a certain range of engineering service. Even glass itself is not denied use because it lacks ductility, its drawbacks are cared for by not putting it where it is expected to carry much static or impact load, and by arranging, in shatter-proof glass, that when an accident does shatter it, the pieces stay stuck instead of flying. A shatter-proof windshield, as a structure, may be classed as non-brittle, though the bread members of the sandwich are still brittle. Consider carburized and nitrided steels. Deformability without irreparable damage can often be engineered into a structure, as by the use of a shear pin in an outboard motor drive. Judicious use of springs, rubber or the like can sometimes be made to serve a similar purpose in guarding against the results of accidental overload. We might more often stop to figure out whether the essential is ductility in the metal parts or deformability in the structure.

We seldom use glass-brittle metals, but we do use some that by contrast with others are of very low ductility, but which are still not entirely devoid of some kind of toughness. We pick and choose our engineering applications of the more brittle materials, making due allowance for their shortcomings. Consider the uses of Duriron.

A shift in the point of view of automotive designers is evidenced by a comparison of the properties demanded in the forged, heat-treated steel crankshafts for the Liberty motor, around 140,000 psi. tensile strength, 120,000 psi. yield strength, 18 per cent elongation, 55 per cent contraction, 50 ft-lb. Izod impact, with the properties of the Ford cast crankshaft, which Russell and Welcker (46) found, on specimens cut from a commercial crankshaft, to be 78,000 psi. tensile strength, zero elongation, zero contraction, 1 ft-lb. Charpy impact. In separate test specimens, around 2 per cent elongation and contraction can be obtained. Millions of Ford cars in operation prove the cast alloy excellent for a crankshaft of the design nowadays used to secure stiffness, but to propose it for a crankshaft at the time it was introduced was rank heresy. How many engineers would have seconded the proposal on the basis of a statement of the properties, with zero ductility figures staring them in the face?

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[2] Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 - 38 T), 1939 Book of A.S.T.M. Standards, Part I, p. 1153.

[3] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this paper, see p. 576.

Malleable iron has a peculiar combination of toughness and brittleness. Notched bar impact tests show only 7 to 10 ft-lb. Charpy, 8 to 13 Izod, on material of present-day ductility. Yet the wedge-curling test shows remarkable toughness (4). The 1915 A.S.T.M. specifications required only 38,000 psi. tensile strength, 5 per cent elongation, and today 40,000 psi. tensile strength, 30,000 psi. yield strength, 5 per cent elongation remain as specifications for pipe fittings, usually made of cupola malleable. The Society's Standard Specifications for Malleable Iron Castings (A 47 – 33)[4] now call for 50,000, 32,500, 10 per cent in one grade and 53,000, 35,000, 18 per cent in another. The 18 per cent grade is often called for in railroad work, but other large users, especially the automotive industries, remain content with the 10 per cent grade. Contrary to the trend with most other materials, as the yield strength of malleable iron rises so does the elongation, hence, if the mere ductility increase confers better engineering usefulness upon the high elongation material, that factor is hard to untangle.

Short cycle or pearlitic malleable, in distinction from regular malleable iron, tends to show a decrease in tensile ductility as strength increases. One may have such figures (5) as 90,000 psi. tensile strength, 70,000 psi. yield strength, 4 per cent elongation. If such material has, in service, the toughness that would be expected from the area under the stress-strain diagram it would seem to deserve wider engineering use than it has yet had. Its relatively slow acceptance may be largely due to the low figure for tensile elongation and the lack of information on behavior under notched conditions.

The case of pearlitic malleable, with twice the yield strength but only about one-fifth the elongation figure of the strong grade of regular malleable iron, is one which well exemplifies the fact that when we have merely the strength and ductility figures of the tension test without intimate knowledge of other idiosyncrasies of a material, we are not in a position to make a sound engineering appraisal of its usefulness.

Ordinary heat-treated duralumin has around 60,000 psi. tensile strength and 18 per cent elongation. In modified compositions 75,000 psi. tensile strength is reached, with 12 per cent elongation. How much do we know from those figures as to what types of service one variety is suited for and the other not?

If ductility figures on transverse specimens were available for all the bar stock that is tested only in the longitudinal direction, our subconscious estimate as to the necessary minimum ductility for acceptable service would be modified. A large proportion of material in service would show shockingly low transverse ductility. Service stresses are not necessarily applied in the longitudinal direction only! If we were to compare true transverse stress with transverse ductility instead of design stress and longitudinal ductility, we would lower our sights on the minimum ductility figure we need to aim for.

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[4] 1939 Book of A.S.T.M. Standards, Part I, p. 499.

Perhaps the consideration of what actually goes on in the tension test may help to indicate what the figures can and cannot show.

THE MECHANISM OF PLASTIC DEFORMATION

Much of the basic information about the mechanism of distortion and fracture in the tension test has been presented in the course of the Society's Symposium on Impact Testing.[5] It was there brought out by Hoyt (6) that ductility is not an independent property but is a manifestation of plasticity which occurs when the shear stresses that produce plastic flow are not accompanied by tensile stresses that exceed the cohesive strength. To be ductile in tension, a metal must have a cohesive strength at least twice its shear strength (resistance to deformation), since the tensile and shear stresses are in a 2:1 ratio. That is, ductility or brittleness in tension is a result of the ratio of the strength properties of the material. This strength ratio is inherent. The ratio of stress can be altered by the shape of the specimen, so that a specimen of the same material can behave in a ductile or a brittle fashion depending on how the stresses are applied.

A notch alters the ratio of applied tensile and shear stresses. Suppose the material has a strength ratio of 2.5:1, cohesive strength to resistance to deformation. Then, in the standard unnotched tension test, with the ratio of 2:1 tensile to shear stress, the specimen is ductile. Surround the specimen with a notch, due to which the stress ratio is 3:1 tensile to shear, then the specimen is brittle. Note that it is the specimen, not the material, that is brittle. To carry this a bit further, we might conclude that, in an annealed or stress-free state the "toughness" or "brittleness" of a metal depends on the ratio of its cohesive strength to its shear strength and the way in which loads are applied.

If ductile or shear type failure starts under stress, the elongation and contraction will depend on

1. The magnitude of the original ratio of cohesive strength to shear strength, and

2. The rate at which this ratio decreases under plastic flow.

Thus, a high value of elongation may originate in two ways:

1. The ratio of cohesive strength to shear strength may be very close to 2:1 in the virgin metal; however, this ratio may change very slowly with plastic flow and failure will thus be delayed until high values of elongation are reached.

2. The ratio of cohesive strength to shear strength may be much greater than 2:1 in the virgin material; however, the ratio may drop more sharply during plastic flow with the result that an elongation similar to that in (1) would be obtained.

While the two materials would thus have the same "toughness" or "ductility" as measured by elongation, it would be expected that they would behave quite differently under fatigue or impact stresses.

Thus, material of identical tensile strength, yield strength, elongation, and contraction on a standard specimen may be quite different in response to tensile stress when they are in nonstandard geometric shape, or under a more complex system of stresses. The brittleness that may ensue when the stress system is that induced by a notch is what is sought to be revealed by notched-bar impact testing, and, historically, Izod developed such testing in order to discriminate between steels which, according to the tension test, were of equal toughness.

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 21 (1938).

The action of the two stress systems and the response of the metal to them can be seen in the fractured tension bars. Materials with low cohesive strength break before plastic deformation can come into play, little elongation or contraction occurs, the fracture is flat and at right angles to the tensile stress—it is a "brittle fracture" as occurs in cast iron. Other alloys, like cast manganese bronze, stretch rather steadily up to fracture and neck down by a gradual taper. Annealed austenitic steels show almost uniform elongation and contraction throughout the gage length. In cast bronzes and in malleable cast iron the numbers for elongation and contraction are usually quite close to each other. In normalized cast steel, the contraction figures normally run from $1\frac{1}{2}$ to 2 times the corresponding elongation figure. In wrought, annealed, normalized, or quenched-and-tempered steels of usual composition and treatment the contraction figure is generally around twice that for elongation; that is, there is marked local necking. The mode of deformation varies in different alloys.

When no appreciable plastic deformation occurs, the fracture starts at the periphery and progresses across the section. When marked necking occurs, the fracture starts at the center. If the test is stopped at this point and the bar is sectioned longitudinally, a flat spot is found where the middle of the bar pulled apart while the periphery still held and was still undergoing plastic deformation. The deformation produces a radial tensile stress at right angles to the applied tensile stress—the center spot is therefore under three-dimensional stress and under such stress the metal, even though ductile, fractures in brittle fashion (7). Finally the outer ring gives way on the 45-deg. plane of the applied shear stress and shows the "cup and cone" fracture. Thus the fractures, ranging from all flat, a large flat surrounded by a small cup and cone, a small flat surrounded by a large cup and cone, to full cup or star, themselves give something of a record of the relative cohesive strength and resistance to deformation of the metal. Differences in mode of deformation may thus show up, even though the total energy to fracture may be the same.

Merit Indices:

Various "merit indices" have been advanced which purported to evaluate the relative weights of the various tensile properties. The merit indices are little more than a feeble attempt to condense into one quantity, from the figures reported in the tension test, but without taking a complete stress-strain curve, some approximation to the area under that curve. Various weightings have been used without making any one index much more useful than another. All the indices labor under the difficulty of comparing apples and pears, and fall short of giving the information of the complete diagram.

Toughness:

It is often true that while we ask for, and measure, ductility, what we are really after is toughness.

The qualitative term "toughness" is broader than that of "ductility." Lead is ductile, but we do not call it tough; it has not the strength implied in toughness. The area under the stress-strain curve is a measure of the energy required to fracture the specimen—*in the tension test*, that is, *where deformation is free to occur* just as it occurs in the standard tension test on the standard specimen. For this condition, that area is one measure of what we term toughness, though one that still has to be viewed in the light of the strength properties. Pure aluminum with low strength but high ductility, and cupola malleable iron of higher strength and low ductility, might show the same area under the stress-strain diagram and by this measure would have the same toughness, but not by other criteria.

Four Cases of Ductility Requirements:

Keeping it firmly fixed in mind that different engineering materials do have different modes of plastic deformation, in order to keep from approving false generalizations that may have been drawn, and recalling also that the measurements made tell only the final condition, after stressing to fracture, but nothing whatever about the early stage of plastic flow, so as to be mindful of the inherent limitations, let us see how the engineer actually uses ductility figures.

There are four cases for which the engineering import of ductility of a metal, as measured in the tension test, may be considered:

1. The metal is not in its final form but is a raw material which is to be shaped into final form by plastic deformation, as in stamping out an automobile fender.

2. The metal is in final form for engineering use, but in normal service is called upon for plastic deformation.

3. The metal is in final form, the engineering service calls for no plastic deformation, and may even forbid any such deformation whatever, but accident insurance is required in that if failure does occur through accident, it must occur by plastic yielding rather than by snapping off.

4. The metal is in final form, the normal service or the accident insurance aspect admittedly calls for relatively little ductility, but a large measure of ductility is likely to be demanded by the engineer because properties which he may not be able to measure or even to define, are needed in normal service, and experience, intuition, or superstition, tells him that by calling for ductility he will probably get the other properties. Here ductility is not the road bed, but merely a sign post along the road.

In all four cases we are confronted by the question of how much ductility to demand, that is, how to draw specifications.

Case 1. Material for Plastic Forming.—In fabrication by bending, deep drawing and the like, ability for large local plastic deformation without attendant embrittlement or, as it is termed, without "exhausting the ductility" is desired. In evaluating suitability for such deformation, a knowledge of the yield and tensile strengths and the ductility properties, especially the transverse ductility, the hardness, the grain size and the propensity toward work hardening, is helpful, as a first approach. Cupping tests, wedge-drawing, and in particular, bend and repeated bend tests also help. Among all of these, perhaps tensile ductility is the guide most depended upon, though the repeated bend test seems worth consideration as perhaps representing a still closer approximation to what we really want to know.

I understand that, at a conference on cold work held in Pittsburgh in the fall of 1939, Winlock, whose opinion deserves great respect, cited tensile ductility as the best single guide to deep drawability. It is, indeed, customary to demand a certain level of tensile elongation as a first requirement, then make a further evaluation on the basis of several other pertinent factors.

But it is so notorious that the only way to evaluate precisely the deep drawing properties of a steel or a non-ferrous alloy for a given job is to try it out on that particular job, that it is not necessary to labor the argument here. We may state categorically that the tensile ductility figures alone are not a satisfactory basis for complete evaluation of deep-drawing behavior.

In this case we do want plasticity, and we want all we can get. The urge for high formability is justified. What we badly need is some more accurate criterion than tensile ductility.

Case 2. Normal Service Calls for Plastic Extension.—Examples under this heading which call for large plastic extension in service are scarce, though some parts might be said to require plastic formability in normal use. A pewter pitcher has advantages over a glass one in that when dropped and dented it can be beaten back into shape. Spectacle bows get out of shape and have to be adjusted. A tin can must dent rather than crack when the filled can is dropped. Fenders must be capable of being uncrumpled from time to time, and axles need to be capable of bending back to bring the front wheels into alignment after road shocks have misaligned them. But the ability to be restored is a virtue only because of an initial failure. If any of these parts had sufficient elastic strength and "crackless plasticity" to resist both permanent deformation and shattering in the first place, there would be no need for the virtue of restorability.

A case where tensile ductility may correlate quite directly with service requirements appears in pipes that may accidentally freeze in normal service. It is found that ordinary lead pipe and pipe from lead containing 0.05 per cent tellurium differ in the number of times water may be allowed to freeze in them before they will burst. On a slow tension test (52), ordinary lead shows some 50 per cent elongation in 8 in., tellurium lead some 90 per cent, and the number of freezings to failure is about in that order. If the tension test is run rapidly, the figures are something like 53 per cent and 56 per cent, respectively, and in rapid freezing the corresponding pipes differ little in number of freezes before bursting. The matter is complicated by the fact that the tellurium also increases the tensile strength, and it markedly confers the property of retention of work hardening, which property is lacking in ordinary lead, so the behavior is not necessarily ascribable wholly to the difference in elongation. However, in copper pipe that must stand accidental freezing, annealed copper with lower strength and higher elongation behaves better than hard drawn.

It should be noted that in these cases, it is an abnormal rather than a normal condition of service that calls for ductility, and that neither the lead nor the copper pipe will stand many repeated freezings. The cases where the need for formability of such degree actually enters into the expected service before failure are decidedly rare in engineering practice. In the stretching of the pipe through the expansion of water on freezing, tensile ductility is exactly the property that is utilized, is a valid direct measure in itself, and the demand for high figures is reasonable.

Ability for Stress Distribution in Static Loading.—A smaller degree of capability for yielding in use is called for in many applications of structural steel. Everyone agrees that some degree of tensile ductility is required in such structures as bridges, in structural framework for skyscrapers, in ships and the like, where a number of stressed members must be joined by bolting, riveting or welding and perfect alignment cannot be secured. There is, however, no agreement on just how much ductility is required. Local yielding is necessary to distribute the stress, so material with some ability for plastic deformation is always used for tension members. The Society's Standard Specifications for Steel for Bridges and Buildings (A 7 – 39)[6] prescribe a minimum elongation in 8 in. of $\frac{1,500,000}{\text{tensile strength}}$ and a minimum elongation in 2 in. of 22 per cent. In the Standard Specifications for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 212 – 39)[7] the minimum elongation in 8 in. for flange grade is $\frac{1,550,000}{\text{tensile strength}}$. For 70,000 psi. tensile strength, these values are 21.5 and 22 per cent elongation in 8 in. In the Standard Specifications for Boiler and Firebox Steel for Locomotives (A 30 – 39)[8], the minimum elongation in 8 in. for flange grade is $\frac{1,500,000}{\text{tensile strength}}$ and for firebox $\frac{1,550,000}{\text{tensile strength}}$. For 60,000 psi. tensile strength, these are 25 and 26 per cent. In the Standard Specifications for Structural Steel for Ships (A 131 – 39)[9] the minimum elongation in 8 in. for structural steel is $\frac{1,500,000}{\text{tensile strength}}$ which for 65,000 psi. tensile strength would be 23 per cent. With the same fraction for rivet steel and steel for cold bending the elongation at 60,000 psi. tensile strength would be 25 per cent.

That is, on such steels of 20 to 26 per cent elongation, a difference of 1 or 2 per cent in the figure is supposed to delineate steels into certain classes for different engineering services. It takes some stretch of the imagination to see why 20 per cent elongation is not ample to allow all the local readjustment necessary to distribute stress, whether it be in a bridge structure or in allowing a ship to "find herself" on her shakedown run.

Aitchison (8) gives a 20 per cent figure in a back-handed sort of way. He says the steel should "be capable of undergoing a certain amount of distortion without fracture." "In other words the steel must possess a reasonable degree of ductility." "The degree of distortion necessary depends on the smallness of the radius involved. If, however, it is considered that the actual portion of the steel part which is to distort is perhaps only 0.01 in. deep and 0.01 in. long, an elongation of 20 per cent in this particular volume of steel will have an inappreciable effect upon the general dimensions of the part." He does not definitely state that 20 per cent is the "certain amount" or the "reasonable degree." In another place he comments that 12 per cent elongation is good for a steel of 170,000 psi. tensile strength, while for one of 100,000 psi. tensile strength it would probably be good only if it were more than 20 per cent. Further on he says it is obvious that no particular value of elongation or contraction is a safe value, or one that will guarantee the life of a part, since "ductility factors are called into play in engineering parts and structures only under very special conditions and circumstances."

Epstein (60) cites Miller as believing that 5 per cent elongation about a rivet hole in structural steel is an adequate amount.

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[6] 1939 Book of A.S.T.M. Standards, Part I, p. 1.
[7] *Ibid.*, p. 60.
[8] *Ibid.*, p. 37.
[9] *Ibid.*, p. 17.

The matter of ductility in welds was argued in a symposium held by the American Welding Society several years ago (10). In this Jennings cited inconsistencies between ductility of welds as measured by tension and by bend tests, Moore pointed out that neither tensile ductility nor bend values measure the ductility needed in "crackless plasticity," Chapman expressed the belief that ductility is useful as a measure of cleanliness, and Greene said that, for welded pressure piping, there is no need to call for more than 10 or 12 per cent elongation, since quality of weld is far more important than ductility, and, anyhow, it is the first 0.1 per cent deformation that is most important.

Ability for Stress Distribution Under Repeated Stress.—Shifting our attention from the structures that are subjected to static stress to machine parts for withstanding repeated stress, some ability for distributing stress is required, but we can hardly call that ability ductility as ductility is ordinarily defined.

A usual explanation for good behavior under conditions of stress concentration and particularly for good behavior in such conditions under repeated stress, is that the metal at a sharp angle, like a bad fillet, at a notch or at the tip of a crack, can undergo very localized plastic flow, thus rounding off the angle and distributing the stress. Some form of something like ductility is postulated. Moore (11) calls the property desired "crackless plasticity," but says (12) that deformation only of the order of 1 per cent over a minute area seems to be involved. He (13) remarks, "the beneficial effect of slight, crackless, plastic action in easing overstress is probably the salvation of many a crank pin and railroad rail." But, as Peterson and Moore (14) pointed out, such readjustment occurs early in the first overload or at least in the first few repetitions of relatively small overload, while tensile ductility measures what occurs throughout a single huge overload—one that is actually carried to failure before the measurement is made. The distortion during the "easing" process is very small. In view of the comment of Moore on railroad rails, we might recall that a rail is not a very ductile or tough material—it may have some 9 per cent elongation, 12 per cent contraction, and 2 ft-lb. Izod.

After a long private discussion of ductility among a few eminent engineers and metallurgists, they agreed that the situation could probably be summed up as follows: "If in a finished machine or structure all the metal is capable of withstanding 1 per cent further elongation, this 1 per cent will be sufficient for stress adjustments. This means 1 per cent along very short gage lengths at locations where there is high stress concentration."

From another point of view a figure of 1 per cent has been arrived at by Rôs and Brandenberger (15), who say that, in the welding of soft steel pressure piping, if the pipe has previously been bent so that an extreme fiber shows more than 1 per cent elongation, the piping should be annealed 2 hr. at 1150 F. to make it stress-free and "overcome the drop in ductility and impact resistance due to cold work." Implied in this is the idea that 1 per cent permanent elongation is not harmful.

Total Flow versus Initial Flow.—From such instances and such comments, the question emerges whether the measured tensile ductility, which includes performance clear up to fracture, has any real bearing on engineering requirements of a part ready for service, and whether the really important factor in plasticity is not the *initial* plastic deformation, the *only* deformation we are going to use. The rest may be superfluous.

If, in forming, we need to use nearly

all the available plasticity, we are then interested in the whole of the plastic range. From the engineering point of view, we may be interested in the superfluous ductility as a reservoir for formability under conditions of catastrophe that may occur, not in use but in misuse, that is, as an accident insurance. But all in all, it appears that in the examples that fall strictly under case 2, the useful ductility is far below the level usually specified, so the justification for the specifications must be sought in other needs. Indeed, in many engineering uses for which ductile materials are commonly specified, there is no appreciable general elongation necessary or desired in service; the design is always on the basis of elastic or fatigue loading below the yield strength. Bolts may stretch, and it is better that they stretch rather than snap, but as long as they do not snap, the stretch is no virtue. It is not pleasant to have the fiddle string snap in the midst of the sonata, neither is it good to have it stretch and get out of tune during the playing. A connecting rod that stretches is not at all acceptable to the automotive engineer. The ultimate in freedom from plastic flow is required by the turbine engineer who wants his turbine rotor to be sufficiently creep resistant against the centrifugal forces to keep its stretch below 1 per cent in 100,000 hr. We certainly do not specify tensile ductility because we expect to use it in such cases.

Case 3. Ductility as an Insurance Factor.—Much of the call for ductility comes from our feeling of need for some sort of insurance against accidental conditions. We would prefer that in an automobile collision, the steering wheel bend out of shape rather than break off and jab the end of a spoke into our chest. We feel that a coupler knuckle on a freight car will sometimes have to withstand a greater coupling speed than the railway rules called for. We want the head of a cold chisel to resist both battering down and spalling off.

Choosing material for a refinery still tube so that when overheated it must bulge and thus give warning of impending disaster, is sound engineering (although we should not forget that, if cheap enough tubes were available that never bulged, but stood all possible degrees of overheating without stretching appreciably, they would be the ones chosen).

How much ductility, in percentage elongation and contraction, is needed to avoid snapping off in an accident? The answer seems to be "some." Energy absorption, rather than mere deformation, would seem to be what is required, so again it is toughness, rather than ductility, that is actually wanted, and the qualitative presence of toughness is more important than the quantitative amount.

Case 4. Ductility as an Indicator Only.—Most engineers freely admit that many engineering uses do not demand, and indeed will not allow, any plastic deformation, but in such cases they nevertheless require a high measure of ductility, solely because it is believed to connote a metallographic structure which in turn insures the presence of some other, often unnamed and not clearly understood, desirable attribute. Experience having shown that good behavior in service accompanies the proper structure, ductility is used as a mechanical indicator of structure, so specifications are drawn to insure that future lots shall have the ductility of the previous lots of good behavior. There is logic in this point of view. Nevertheless to be thoroughly logical in it, should the specifications not be drawn so as to reject material above as well as below, the ductility level of the material of past experience? Is it not possible that some of the wanted, but unnamed, attributes are inversely related to ductility?

Is not the selection of ductility as the criterion for other features that are needed something like the assumption that, since some individual has been able to approve all the New Deal nostrums, he is thereby fitted for any Governmental post, from the Supreme Court down?

Some specific cases of the use of ductility as an indicator may be cited. One such use is in the study of stability of cast heat-resistant alloys. An alloy of the 25 per cent chromium, 12 per cent nickel type, as cast, may show say 12 per cent elongation, but after a 48-hr. exposure at 1600 F., the elongation may drop to 3 per cent. Whether one thinks that 3 per cent is enough for the required service of heat-resisting castings or not, he cannot but be disturbed by the change and fear that in high-temperature service a condition might be reached that, by any criterion, would be called brittle. The situation would not be much different if the figures were 40 per cent and 10 per cent, for it is the change rather than the absolute value that is disturbing.

Discussion rages on the "embrittlement" of carbon-molybdenum steel for high-temperature use, based on decrease in static ductility in laboratory tests at operating temperatures, but at loads considerably above design loads. Whether these changes will occur under service loads after very long periods of time or whether they are related to a different mode of deformation under a higher rate of strain than is exerted by loads that are permissible from the point of view of creep, remains to be seen. At any rate, experimenters use static ductility as an indicator in such cases. Often the real concern is not so much the low elongation in itself as is the development of intergranular fissures and the fear of brittleness under shock or repeated load, due to the presence of a sharp notch, irrespective of the ductility of the surrounding metal.

The Bridge Wire Case.—Fissures appeared to be a prime cause of failure in the heat-treated wires of the Mt. Hope and Ambassador bridges (16). The average tensile properties of the usual cold-drawn wire, which does not fail, and of the heat-treated wire, compare as follows:

	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 4 in., per cent	Contraction, per cent
Cold drawn......	223 000	183 000	6.6	40
Heat treated.....	223 000	190 000	6.1	37

However, when a large number of specimens of the heat-treated wire were tested, with their galvanized coating on, occasional specimens with less than 2 per cent contraction were met. When the coating was removed and the surface of the wire cleaned up by reducing the diameter from 0.195 in. to 0.165 in., there were no cases of low ductility. Swanger and Wohlgemuth (16) say that the low contraction was due to defects in the outer layer of the wire which, in the tension test, modified the mode and degree of deformation at the point of fracture. The brittle fractures that occurred in the bridges are ascribed to two causes, pits and lack of seating.

The breaks were fatigue failures, whose nuclei were located at surface imperfections, arising from pits on the wire rod and not "ironed out" by the one or two drafts of the drawing process used on the heat-treated wire, whereas in the five drafts of the cold-drawn wire, they were ironed out. The pits were filled with the iron-zinc alloy layer, and cracks, which started at these points under fluctuating load, progressed through the ductile material beneath.

By study of the stress conditions it was concluded that another point of difference was that the yield strength of the cold-drawn wire, though only 7000 psi. lower, was nevertheless enough lower to permit a desirable plastic flow or creep that permitted seating of the loops and thus lowered the imposed bending stress. From this point of view the really necessary ductility was not that measured in the tension test, but only that first tiny increment of ductility comprised in room temperature creep, and it was equally necessary that this amount of plasticity be available at not too high a load. (We shall refer to this again.)

However, as Thum pointed out in discussion, brittle tensile fractures were obtained now and then at low loads in laboratory tests by incremental dead loading of the heat-treated wire, which tests, had they been given sufficient weight, might well have brought about changes in the manufacturing process which would have avoided the surface pits and made the use of heat-treated wire a triumph instead of a disaster. The lesson taught by tensile ductility was not sufficiently taken to heart.

The interpretation of all the facts about the bridge wire is still in dispute, but the conclusion is inescapable that the invariably good tensile ductility figures for the core of the heat-treated wire did not make the wire immune to the embrittling effect of surface stress raisers.

Guns.—A big gun requires resistance to the shock of firing. All arsenals study the radial ductility of their gun blanks and take great pains to get radial ductility of the same order as the longitudinal. Yet, in absolute figures, the ductility does not have to be exceptional. Dickson (17) gives, for a cast, autofrettaged gun, soaked at 570 F. after the working, 115,000 psi. tensile strength, 90,000 psi. yield strength, 5 to 11 per cent elongation, 36 to 48 per cent contraction. Present values run 115,000 psi., 95,000 psi., 17 per cent and 60 per cent. By various types of shock tests, especially tension impact, the arsenals assure themselves that notch brittleness is absent. If that absence is assured, a relatively small amount of tensile ductility, in relation to that ordinarily required by specifications for steel of these tensile strength properties, suffices.

Such values for gun material make one wonder why 20 per cent or more elongation is needed in a statically stressed bridge, and emphasize the fact that, broadly speaking, ductility specifications for material other than that to be fabricated by plastic forming are imposed primarily to obtain something else than ductility.

Cast Steel.—A case where stringent requirements for static ductility are imposed is that of cast steel. In the Society's Standard Specifications for Carbon-Steel and Alloy-Steel Castings for Railroads (A87 - 36)[10] the following properties are specified for grade B steel: 70,000 psi. tensile strength, 38,000 psi. yield strength, 24 per cent elongation in 2 in., and 36 per cent contraction. While impact values are seldom specified, in general, a steel meeting or exceeding this ductility shows over 25 ft-lb. Izod impact, one failing to meet it less than 25 ft-lb. Commercial cast steels generally run 75,000 to 80,000 psi. tensile strength, 45,000 to 50,000 psi. yield strength, the strength requirements are easily met, but much difficulty has been encountered in consistently meeting the ductility requirements. When making steel castings that were aluminum treated to avoid gas porosity Sims and Dahle (18) very definitely correlated the failure to meet ductility specifications with the presence of inclusions as films at crystal boundaries. When slightly larger amounts of aluminum were used and the sulfur content controlled, the inclusions became globular and the ductility and impact requirements were consistently met.

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[10] 1939 Book of A.S.T.M. Standards, Part I, p. 224.

Here a hair-trigger difference in ductility values is an indicator of a real difference in the metallographic make-up of the steel. That the values in themselves, as numbers, mean anything from the engineering point of view is very doubtful. If both steels were sound and homogeneous, in what engineering use could one differentiate the behavior of say, steel A of 22.5 per cent elongation, 27.5 per cent contraction, and 22.5 ft-lb. Izod impact and steel B of 24 per cent elongation, 36.5 per cent contraction, and 28 ft-lb. Izod impact?

The justification for the specification and for the rejection of steel A, if they are just, rests not in those figures, but in the fact, commented on by Frank in discussion and also by Gagnebin (**19**) that the "low" ductility specimens generally show irregular, woody fractures, that is, that the film type of inclusion affects the mode of deformation and introduces a factor of uncertainty. What it is really desired to guard against is this uncertainty. The ductility is mere indirect evidence of the metallographic structure. Yet the specification, couched only in the language of tensile ductility, seems to bar perfectly good steels.

This is more obvious when the requirements are stepped up to those of the Society's Standard Specifications for Alloy-Steel Castings for Structural Purposes (A 148 – 36)[11] which give for class B, grade 2 steel 90,000 psi. tensile strength, 60,000 psi. yield strength, 22 per cent elongation, 45 per cent contraction, and, implied, an Izod impact value of at least 20 ft-lb. This requires some alloying element, usually between 1 and 2 per cent manganese. Grotts (**20**) reports a 1.85 per cent manganese steel that, after proper heat treatment, gave 110,000 psi. tensile strength, 63,000 psi. yield strength, 22 per cent elongation, 44 per cent contraction, and 26 ft-lb. Izod impact, which is not acceptable under the specification by 1 per cent on contraction. The specified properties can be met and exceeded by the use of other alloying elements in place of part of the manganese and by employing some grain-refining addition. But by what process of reasoning or test do the specification makers arrive at a proper basis for rejection of the 44 per cent contraction, plain manganese steel, free from film-like inclusions or woody fracture? Is it so impossible or impractical to specify microscopic structure or type of fracture that we have to hide what we want behind what we do not need?

DIRECT *Versus* INDIRECT TESTS

All that the tensile ductility figures themselves can do is to lead us to one alcove in the library; they do not tell us on which shelf the wanted book is.

Assumption that there is a causal relationship between tensile ductility and toughness is the lazy man's approach. The figures are cheaply available as a by-product of getting the really essential strength values. To work out specific tests for evaluation of toughness under the loading conditions of service, with restraint of plastic flow and the prevalence of triaxial loading, is a trying job. Only when it is done can we test in the laboratory and rely fully on the indication of the tests. We are finding now that given ductility values may go hand in hand with true toughness under some conditions, and shocking brittleness under others. The signpost may be a Halloween sign; it does not always point in the right direction. When it does, it does not tell what further turns the road takes before the destination is reached. The mere values for tensile ductility directly signify the ductility in the tension test. Any other significance is indirect, often extremely indirect.

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[11] 1939 Book of A.S.T.M. Standards, Part I, p. 230.

This has already been adequately summed up by Russell (21) who says "The numerical value of the elongation and of the reduction of area seldom appear in engineering formulas. These quantities are considered to measure ductility, and no doubt theory will show the desirability of ductility in a structure, but how much is necessary and the most suitable balance between strength and ductility, theory has not told us. But often experience has. That is, we learn from experience that a certain elongation is desirable in material for a certain particular use, or stated differently, we learn that a certain material is suitable, and in selecting subsequent material we want identity in the significant property of elongation. But there is no certain reason to believe that elongation really measures the desired property. In material of another class some other degree of elongation may, and probably will, be better."

When experience is lacking, tensile ductility is no infallible guide. Just as we do not expect an Everglades guide to know the Maine woods, so we should not expect too much from ductility figures outside of a known environment.

In general we have been too reluctant to spend money, too lazy to spend time, or too ignorant to know how to develop direct tests for the actual properties we should be measuring.

The Navy tests armor plate by shooting shells at it, not merely by making a tension test. Whenever we must know the whole truth from a test procedure, we, too, had best use some more direct and searching test rather than to rely too implicitly upon the very limited significance of the ductility figures of the tension test.

SPECIFICATIONS

The group of eminent engineers and metallurgists whose private discussion has previously been referred to, came to another conclusion, not wholly consistent with the one previously cited. This second conclusion is "It is well to specify the highest elongation of a metal which, without excessive cost, can be obtained without excessive sacrifice of other desirable properties; however, it is no indication that because one can get structural steel of 30 per cent elongation, 30 per cent should also be specified for brass and aluminum."

I do not believe it is sound specification writing to demand, without qualifications, a definite figure just because we can get it, without knowing that the engineering requirements actually need that figure.

We should be very careful about what we say in specifications. Whenever we flatly demand particular percentages of elongation and contraction, as in the cast steel case mentioned above, both the supplier and the purchaser tend to take those figures as gospel truth. A barrier very difficult to penetrate has been set up against experimentation with allied materials of a slightly different range of properties. The allied materials may be exactly as good for the engineering use in prospect, cheaper, and capable of greater uniformity in production, but the specification bars the incentive to try them out.

I should like to see footnotes to specifications explaining why the stipulated figures were stipulated, citing chapter and verse to the investigations or records of accumulated experience to justify the values, or else honestly stating that it is not certainly known that the values adequately measure the attributes really needed, that the values were picked out of thin air because the suppliers said they could meet them and the purchasers said they sounded all right. Then, if some one works out a different material, really superior in service, but not meeting a figure for one requirement that has been blindly set up for earlier materials, potential users will not refuse to try the new on the basis of reference to a specification which they erroneously assume was drawn on the basis of proved necessity.

In the foreword to the A.R.E.A. specifications[12] for alloy steels for bridges we find the following:

> "Nickel and silicon steels are excellent bridge steels, but it must be remembered that in every high-strength steel, whether high-carbon or alloy, some desirable property must be encroached upon to secure the added strength. While such steels have ample plastic action between yield point and ultimate strength, it is not as great as for carbon steel. While they have excellent ductility and bending properties, those properties are not quite so good as in carbon steel. Therefore, when such steels are used some effort should be made to compensate in the design and details for these differences. For that reason, the allowable unit stresses in these specifications are a somewhat smaller fraction of the specified yield point than those for carbon steel and slightly greater edge distances are desirable.
>
> In considering other alloy steels as they are made available, tests should be made to develop the effect of rivet holes and riveting, before determining the allowable unit stresses. No steel should be considered satisfactory for bridge construction if its yield point exceeds 70 per cent of the ultimate strength."

Now from manufacturers' data cited by Cone (22), and test data cited by Collins and Dolan (23) it may be seen that Corten, Hi-steel, Yoloy, R.D.S., and H.T.-50 have yield ratios of from 70.5 to 80. I imagine that five metallurgists representing the producers of these five tough low-alloy steels could produce convincing evidence that these steels are not unsuitable for bridge construction, and hence that the last sentence quoted totally fails to express the real intent in the minds of the engineers who drafted it.

Moisseiff (24) says that cold-drawn bridge wire, a material of low ductility, "is the most dependable element in bridge construction." He cites bright cold-drawn wire as behaving all right, save in resistance to corrosion, then goes on to say that, besides providing corrosion protection, hot galvanizing has an annealing action which raises the elongation from 2 per cent for the bright, to 5 per cent for the galvanized wire, and remarks, "this increase in elongation is of great importance in a material so 'short' in behavior."

One must accord great respect to Moisseiff's opinion, but one does wish he would elaborate as to just why this "most dependable" material needs to be given greater ductility.

ALLEGED CORRELATIONS WITH DUCTILITY

What are some of the factors, unnamed in the usual specification, that are sought through the ductility figures? And how do they correlate with ductility?

An attempt is sometimes made to correlate the tensile properties, especially contraction, with machinability. Thus the accepted belief that an addition of molybdenum to an alloy steel to be heat treated often allows such steel to be machined at a very high hardness level, has been ascribed to a slightly greater contraction shown by the molybdenum steel at that hardness. However, belief that this relationship is a necessarily causal one is very much weakened by the finding that the introduction of a little lead into an alloy steel of high hardness greatly facilitates machining but leaves all the tensile properties, as ordinarily determined, including elongation and contraction, unaffected.

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[12] Specifications for Steel Railway Bridges: III. Alloy Steels, *Proceedings*, Am. Railway Eng. Assn., Vol. 36, p. 681 (1935).

Fine-haired differentiation on the basis of static ductility is often used as an argument in favor of one or another alloy steel for heat treatment by quenching and tempering. If such comparison is made on the basis of treatments that give equal tensile strengths and, further, on cross-sections that harden clear through, so that the test values do not relate merely to the surface, it is noteworthy that there exist only such minor differences in yield strength and ductility (as has been brought out by Clapp (25) and French (26)) that an engineering choice among them, on the basis of tensile properties alone, is difficult. A large number of steels might be regarded as interchangeable. Strauss (27), Grossmann (28), and Boegehold (29) all point out, however, that for a given level of tensile strength, yield strength, and elongation, there are measurable differences in contraction. But, as Boegehold, Graves (30) and Peterson (31) all bring out, the commercial choice is made on the scores of cost, uniformity, machinability, warping propensities, etc., rather than on any lack of interchangeability from the engineering point of view that can be related to variations in tensile ductility.

Knowlton (32), discussing axle shafts, concludes that behavior under stress concentration is of more importance than mechanical properties, including ductility, while Almen and Boegehold (33), discussing rear axle gears, put resistance to overload in fatigue as of more importance than tensile properties or impact toughness. Riegel and Vaughn (34) reported a correlation of tractor gears of unsatisfactory performance with abnormally low Charpy impact values, though presumably the service conditions and type of failure differed from those just mentioned. Notch sensitivity under repeated stress is appraised as of major importance in aircraft materials both by Moore (35) and by Johnson (36).

At first sight it would appear probable that tensile ductility, notched-bar impact resistance, and freedom from notch sensitivity in fatigue, all should be very definitely related. It is assumed that ability to deform in tension, notch toughness and "crackless plasticity" are pretty much synonymous. The assumption is not justified.

It will be recalled that equality, as shown by the standard test, in tensile strength and ductility and hence in area under the diagram does not insure equality in toughness under other conditions of test, in respect to "temper brittleness," and in respect to the shift of notched impact resistance with temperature. At this shift from tough to brittle fracture in impact, the static strength and ductility do not show a corresponding discontinuity. McAdam and Clyne (37) state that "as a measure of ductility and toughness (with the exception of cold brittleness) much importance should be assigned to the reduction of area." "In the absence of cold brittleness this property is a good index of the impact value obtainable." However they also go on to say that "much effort has been wasted in aiming at specific impact values" and emphasize the greater importance of determining whether the impact-temperature curve shows a break, rather than worrying about the absolute magnitude of the numerical impact value.

Riegel and Vaughn (34) supply confirmation of both points of view, that contraction is ordinarily a measure of impact resistance, but not necessarily so. They cite two heat-treated $3\frac{1}{2}$ per cent nickel steels of practically identical compositions, similar tensile strengths, around 280,000 psi., and exactly equal yield strengths, 214,000 psi., and each with an elongation of 11.5 per cent, but otherwise varying as follows:

	Contraction, per cent	Charpy Impact, ft-lb.	Notched Tension Impact, ft-lb.
Steel A......	44.3	18	62
Steel B......	34.1	5.5	9

But they also cited another pair of steels, each with 295,000 psi. tensile strength, equal yield strengths and 12 per cent elongation, which gave

	Contraction, per cent	Charpy Impact, ft-lb.
Steel C.........	45	12 to 16
Steel D.........	45	2 to 4

and say: "This indicates the futility of attempting to predict notch-toughness from the area under the strain curve."

Even when single-blow notched-bar impact figures are in the direction expected from the relative ductility, the notched endurance results may go in the other direction, as Bacon (38) shows by the following comparison:

Steel	Tensile Strength, psi.	Yield Strength, psi.	Elongation, per cent	Contraction, per cent	Brinell Hardness	Izod Impact, ft-lb.	Endurance Limit, Polished, psi.	Endurance Limit, Square Shoulders, psi.
0.55% C...............	124 000	65 000	21	44½	245	5	62 000	28 000
0.30% C 4.60% Ni .... quenched and tempered	124 000	108 000	25½	59	254	71 to 86	67 000	24 000

The higher ductility of the second steel did not help its notched endurance. But note the difference in yield strengths; we shall refer to them later.

These comparisons are on rather hard steels; the point might be raised that the area under the diagram ought to correlate better with other criteria of toughness in other classes of steels.

Collins and Dolan (23) present a wealth of data on a few hot-rolled soft steels, including ordinary structural (0.21 per cent carbon) steel, a low-manganese chromium steel of twice the tensile strength of structural steel, one steel of about the same carbon content as the structural steel but containing nickel and copper, and two of the low-carbon, high-phosphorus, members of the high yield strength, low-alloy steel group. Data are provided on hardness and (using standard specimens) on yield strength, tensile strength, elongation in 8 and 2 in., contraction, and area under the stress-strain curve. Static tension tests on notched bars; tension-impact on smooth and notched bars; Charpy impact tests; axial endurance tests from zero to maximum tension on smooth bars; rotary endurance tests on smooth bars, on square-notched bars and on smooth bars with a drilled hole, as well as corrosion-fatigue tests in water on smooth bars, are all included.

All the materials would be classed as ductile, with elongation in 2 in. ranging from 23 to 42 per cent and contraction from 55 to 72 per cent. Tension-impact strength on smooth bars correlated reasonably well with the area under the static stress-strain curve, but no type of notched-bar test showed more than the roughest type of correlation either with the area under the curve or the ductility figures by themselves. One would have to be very bold to predict the notch toughness or notch sensitivity of these steels from the ductility values of the standard tension test.

It is sometimes assumed that good or

poor behavior in single blow notched-bar impact tests means correspondingly good or poor behavior under repeated stress in the presence of stress concentration. Inglis (39), however, has given data that effectively disprove the assumption. He studied a temper brittle nickel-chromium steel that had become embrittled through several years use at 750 F. Its room temperature single blow notched-bar impact value had dropped from the original 38 ft-lb. to 4 ft-lb. Yet endurance tests on threaded specimens gave exactly the same endurance limit, 23,500 psi., for the original and the embrittled states. The "crackless plasticity" called upon in notched endurance had not deteriorated, though the notched impact value had seriously deteriorated.

In ordnance, steels with high ductility in the static tension test may be temper-brittle, as shown by tension impact tests and corroborated by failure of guns in firing tests.

The type of ductility necessary for stress adjustment under repeated stress is so important a matter that we should examine in some detail the idea which everyone is likely to have on first approaching the subject, that is, that high tensile ductility, especially high contraction, which should measure ability for local plastic distortion, is required to insure ability to distribute the local overload. It is true that steels of low ductility do show large degrees of stress intensification, but it seems at least as accurate to omit reference to ductility and just say that notch sensitivity in fatigue increases with hardness or strength. Karpov (40) indeed plots the percentage of endurance strength, as determined on mirror polished bars, that is lost due to a sharp circular notch, as a straight line running from 15 per cent on a soft steel of 40,000 psi. tensile strength to 65 per cent on a hard steel of 220,000 psi. tensile strength.

Indeed, just as no good correlation has ever been found between endurance of smooth bars and tensile ductility, there is equal failure of the more-to-be-expected correlation between notched endurance and ductility. One needs only to recall the low notch sensitivity of cast iron.

An extruded, 8 per cent aluminum, 0.5 per cent manganese, balance magnesium, alloy of say 47,000 psi. tensile strength, 35,000 psi. yield strength, 12 per cent elongation, according to Wood (41) will have an endurance strength on a smooth specimen for 500 million cycles of about 16,000 psi. Sachs (42) reports 23,000 psi. at 10 million cycles. With a collar about the specimen, which is equivalent to a square notch, the 10 million cycle figure drops to 7000 psi. That is, magnesium alloys are markedly notch sensitive in spite of appreciable ductility.

Templin (43) points out a general trend in aluminum alloys for more deleterious effects of notches in fatigue as the static tensile elongation decreases, but points out emphatically that "the numerical values of the elongation *per se* cannot be used as a satisfactory basis for predicting the effect of notches on the fatigue strength of the various alloys in all their different conditions."

Lundgren (44) has plotted the endurance ratio (endurance limit for polished bars divided by tensile strength) against percentage contraction for a large number of steels, to uphold his contention that a high endurance ratio and high contraction go hand in hand. He is forced to break the steels down into several classes to accomplish any sort of correlation, and even then the plotted points remind one of a poor pattern from a 12-gage gun. Moreover, while the ratio of endurance limit of polished bars to tensile strength is an interesting academic figure, the absolute level of the endurance limit in pounds per square inch speaks more loudly in engineering applications. Similarly the notch sensitivity ratio (endurance limit on polished bars divided by endurance limit on, say, square-notched bars) or the percentage drop of the notched from the polished figure, often serves to make us uneasy about a steel that shows a large percentage drop, although what it is a percentage of may be large enough so that the final product, the notched load-carrying ability in pounds per square inch is better than that of a less notch-sensitive comparison steel.

Even if it were true that high contraction insures a high polished bar endurance ratio, there is no proof that a high polished bar endurance ratio connotes resistance to notch formation or propagation, or a high absolute level of notched-bar endurance limit.

Whether we discuss a limited group of like steels, or whether we compare alloys of wholly different chemical composition, that is, those of different metals as bases, the same situation is met—possible qualitative indications toward a not-too-bad notch sensitivity in highly ductile alloys, but an entire lack of quantitative correlation.

Damage:

With the expectation denied that tensile ductility defeats a notch, one might then think that perhaps if a crack is self-formed during repeated stressing, instead of some external stress-raiser being imposed, tensile ductility may measure either or both the resistance to formation of a crack and its propagation when once formed. Though data on "damage line" endurance testing are scanty, the work of French (45), of Russell and Welcker (46), of Russell (47), and of Kommers (48), is sufficient to show an equal lack of correlation. Russell's article reports results on cast steel, malleable iron, and Ford crankshaft material, all containing enough copper to produce precipitation hardening effects. When the precipitation hardening heat treatment was applied it raised the yield and tensile strengths and measurably, though slightly, decreased the elongation and contraction of the steel and the malleable iron, while the tensile strength and the (zero) ductility of the Ford alloy were unchanged. The notched endurance limits were not lowered by the precipitation treatment. Thus a depreciation in tensile ductility did not depreciate the notched endurance.

In the more ductile or non-precipitation-hardened condition, none of the three alloys was able to resist overstress on a polished bar. The "damage lines" lay at the levels of the respective endurance limits. After the precipitation hardening treatment the damage lines, especially for the notched bars, were so much lifted that they became almost coincident with the *S-N* curve itself; the precipitation-hardened materials were made notably resistant to damage by overstress. This improvement would not have been predicted on the basis of the tensile ductility; rather the reverse would have been expected.[13]

Wishart and Lyon (50) determined the endurance limits and the damage lines, using smooth specimens, at a range of temperatures down to −70 F. for three steels, a normalized S.A.E. 1020, relatively weak but with 25 per cent elongation, and two stronger hot-rolled steels with 12 and 16 per cent elongation. All three steels, at room temperature, showed some resistance to damage by overstress; that is, there was a gap between the damage line and the *S-N* curve for virgin material. This gap was of about the same general size and shape at any test temperature for the two less ductile steels. On the most ductile, S.A.E. 1020, however, the gap closed up with decreasing temperature and at −70 F. came quite close to the virgin *S-N* curve, denoting increased ability to stand overload without damage. Wishart and Lyon tentatively ascribe this to the greater ductility of the S.A.E. 1020, and point out that tensile ductility decreases as the temperature drops. In the case of this ductile steel, the steel gets more able to resist repeated overload as it gets colder and less ductile.

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[13] French (49), however, found small resistance to damage in a high-carbon, copper-silicon steel after a combined quench, temper, and precipitation treatment which hardened to 418 Brinell and gave it an endurance limit of 110,000 psi. on an unnotched bar. No damage data are available for comparison on such a steel without the copper.

Russell compared the damage behavior, both on smooth and on notched longitudinal and transverse specimens of two wrought irons, one of which had equal longitudinal and transverse ductility, the other showing some five times as high ductility figures in the longitudinal as in the transverse direction. The notched endurance limits, in the four tests, longitudinal and transverse on both irons, all ran between 18,000 and 20,000 psi. On damage line tests, all were quite insensitive to overstress save the longitudinal tests of smooth specimens of the first-mentioned iron, the one that was equally ductile in both directions.

On the other hand, ingot iron, with higher tensile ductility than either of the wrought irons, even on longitudinal specimens of the latter, also gave 19,000 psi. notched endurance limit but, both on polished and notched specimens, was badly damaged by overstress.

Kommers (48) made a very extensive study of the effect of overstress on three steels and a cast iron. Two of the steels had the following properties:

Steel	Tensile Strength, psi.	Yield Strength, psi.	Elongation, per cent	Contraction, per cent	Endurance Limit, Polished, psi.	Endurance Limit, Notched, psi.
Y......	123 900	66 700	17	30.4	51 200	24 600
X......	134 500	64 800	14	20	48 800	30 400

Overstress tests on polished specimens showed steel X to be decidedly less damaged than steel Y, and while on a percentage basis the damage to overstressed notched bars was greater for steel X, if the data are judged on the basis of the endurance limits of notched bars after a given overstress schedule, steel X is equal or superior to steel Y. Certainly the higher tensile ductility figures for steel Y do not connote superior resistance to the damaging effect of a notch, or of overstressing.

It seems necessary to conclude that brittleness under conditions of stress concentration, either under impact or under repeated stress, is not necessarily avoided by the presence of even very generous amounts of tensile ductility.

Crackless Plasticity:

From the point of view of actual failures of engineering parts, it appears that fatigue is the mode of failure most to be feared and that this may be what is in the back of the specification maker's mind in setting ductility figures.

Even where engineering design is made on the assumption that a part is to operate under purely tensile stress, axially applied, such cases are hard to find in actual service. Hoisting cables, cables for bridges and the like, work under primarily tensile but also under vibratory stress from other directions. A fiddle string is under tension at rest and under vibratory load in operation. A connecting rod or a sucker rod is under repeated tensile stress, ostensibly axial, but actually not likely to be truly axial. Stiff eyebar tensile members presumably have more nearly pure tensile loading, and bolts operate under loads that are chiefly tensile, but who would like to guarantee that even these are not eccentrically loaded? In all these cases conditions at surface and edges are likely to produce stress concentration and cause material departure from axial loading. The parts are vulnerable to the causes for fatigue failure, that is, progressive failure from a local nucleus.

The primary requirement of good engineering behavior really seems to boil down to what Moore has termed "crackless plasticity." This refers to plastic deformation on a scale so minute that the part does not measurably change in external dimensions, but nevertheless sufficient to bring about a redistribution of stress and to produce, as in strengthening by understressing, many of the results that are produced by the bodily cold working of large plastic deformation.

Offhand, the tension test seems to be silent on crackless plasticity. I wonder whether this is necessarily the case.

Curved Stress-Strain Diagrams:

Is it not possible that the tension test is trying to tell us, or at least to hint to us, more than we hear when we listen only to the elongation and contraction figures, or even to the figure for the area under the stress-strain curve? The curve, if we determine it precisely enough, gives us the modulus, the yield strengths for several amounts of permanent set (the shape of the curve below the yield strength as we usually define it), the slope of the curve while necking is going on, and the dip in the curve as contraction occurs. May not the early stages of the tension test give us information about crackless plasticity?

The process of equalizing and distributing stress through localized plastic deformation, seems to be, on a tiny scale, the same sort of process that we recognize in the bending of a bar. If the extreme fiber of the bar is stretched beyond its yield strength, stress is redistributed. If the stress-strain diagram is markedly curved from the origin, as it is with cast iron on the first application of load, so that Hooke's law is only roughly approximated, the neutral axis shifts significantly as the load increases; that is, stress is redistributed. It would seem that, since service overstress of an intensity that cannot be borne without crumpling up the part is immediately corrected by redesign, once the first design has been put into service and found to fail, we are actually primarily concerned with overstresses of *small* magnitude, that is, those between the endurance limit and the yield strength, whose damaging effect is only shown after many repetitions of stress. The greater the curvature of the stress-strain curve below the yield strength, the less intense need be the overstress to start redistribution of stress.

The seating of the bridge wire loops is a case in point. For this view we also have the fact that cast iron, with a markedly curved diagram from a very low load, has great insensitivity to external notches. Also that we can "ductilize" an aged deep-drawing steel that has succumbed to "warehouse embrittlement" by a skin pass or roller leveling which puts a marked curvature into the diagram below the yield.

On the contrary, we have the view that cast iron is already notched so much internally by graphite flakes that another external notch does not matter. Or, one may say that the behavior of cast iron is due to its high damping power and consequent absorption of energy, a phenomenon not too foreign to distribution of stress, as it amounts to a subtraction of stress. But magnesium also has phenomenal damping power and is notoriously notch sensitive. Magnesium alloys have a curved stress-strain diagram at low stresses so any expectation of good behavior being *inevitably* connected with this type of stress-strain curve is not borne out. Still other factors must come in.

Yet is not the usefulness of the aluminum-silicon alloys for welding of other aluminum alloys, their freedom from hot-shortness, ascribable to the fact that the aluminum-silicon alloys start to flow a trifle at very low loads, as well as have fairly high tensile ductility, so that the cooling joint may give *easily*, as well as just give? Is it not possible that the reason why a spring steel, notch sensitive though it is, nevertheless stands extremely high loads, as service loads go, is because it can give a bit at not very far above the normal design load? Hankins and Ford (51) mention a silicon-manganese spring steel of 250,000 psi. tensile strength, 242,000 psi. yield strength, 186,000 psi. "proportional limit," 5 per cent elongation in 8 in., 27 per cent contraction, and 11 ft-lb. Izod impact. Which is the reason that the spring, though on the brittle side, is not glass-brittle, the ductility or the shape of the stress-strain curve, or both?

That quenched, untempered steel shows a curved diagram but has dangerous brittleness in service, seems to indicate that the internal stresses bring in an overbalancing factor, so that it is hardly fair to ascribe the behavior purely to lack of ductility or to condemn a curved diagram in any alloy from this one example.

That the shape of the stress-strain diagram below the yield strength is intimately related to behavior under repeated stress is indicated by the work of Memmler and Laute, cited by Kommers (48). Repeated understressing or repeated slight overstressing (in respect to the endurance limit) of a low-carbon steel is shown to alter the stress-strain diagram by eliminating the yield-strength jog and producing a curvature quite like that produced by a skin pass or by roller leveling. In the process of strengthening by understressing, this change in stress-strain relations is brought about without altering static elongation or contraction.

That is, the static relations responsive to what goes on under endurable repeated stresses are those between stress and very minute strain below the yield strength. If overstressing is very severe, that is, far above the damage line, then a decrease in elongation can be noted when the overstressed piece is subjected to the tension test. Conversely, it appears that the static properties of interest in picking material for service under repeated stress include the very early stage of plastic flow rather than the advanced stage corresponding to the ductility figure measured in the tension test.

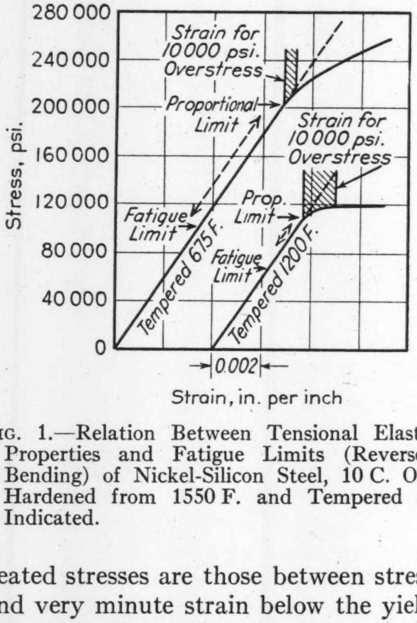

FIG. 1.—Relation Between Tensional Elastic Properties and Fatigue Limits (Reversed Bending) of Nickel-Silicon Steel, 10 C. Oil-Hardened from 1550 F. and Tempered as Indicated.

Hauttmann (**58**) argues in favor of a low "elastic limit" and cites as a virtue in the Gutehoffnungshütte, low-alloy steel, its low "elastic limit" after normalizing from a sufficiently high temperature, which allows it to deform easily in welded assemblies. He points out that with a yield strength (0.005 per cent set) of only 25,000 psi., the endurance limit is 46,000 psi.

The suggestion that in those features of the tension test which have to do with the first occurrence of plasticity there may lie hidden information really pertinent to engineering worth, is by no means new. French (**49**) shows the accompanying Fig. 1 in a discussion of the reasons for an endurance ratio of 49 per cent for a particular steel in a hard condition, and one of 61 per cent on a softer condition. Föppl (**53**) also focuses attention on the ratio between elastic deformation and plastic deformation, much as French's illustration does, in order to differentiate among static and dynamic toughness and brittleness.

The Elastic Modulus:

If we need to compare elastic and plastic deformation, we cannot lose sight of the elastic modulus.

Perhaps a reason why cast iron, zinc-base die castings, plastics and the like are not glass-brittle in spite of low tensile ductility may be their low moduli. Stress distribution by the production of a greater bearing area, say on a gear tooth, could be obtained by sufficient elastic deformation at each load application as well as by plastic deformation at the first application.

That the modulus enters into the general question of stress distribution is nicely shown by von Heydekampf's (**54**) citation of a case where a steel key between keyways in a shaft and hub could not deform sufficiently and produced high local stress and a rubbing action in the keyway of the shaft. When the key was made of copper, the low modulus allowed better stress distribution and failures were avoided.

Submicroscopic particles of copper were, of course, scattered throughout the metal as a result of the precipitation-hardening treatment in Russell's (**46, 47**) tests on copper steel, copper malleable iron and the Ford crankshaft alloy. The presence of these low modulus particles may provide internal cushions for redistribution of stress, since the resistance to damage by repeated overstress was very marked in these precipitation-treated alloys.

Work Hardening:

Another factor that cannot be neglected is the propensity toward work hardening, indicated by the slope of the stress-strain curve during the period of elongation. Russell showed that an annealed 18 per cent chromium, 8 per cent nickel stainless steel was scarcely damaged in fatigue by long-continued repetition of relatively large overstresses and that the notched-endurance limit was above that of an unnotched bar, doubtless because of work hardening during machining of the notch. McAdam and Mebs (**55**) show how the stress-strain diagrams for 18-8 alloy at different degrees of work hardening, persist in showing curvature, and how even severely work-hardened metal is still able to yield a trifle, even at low loads. The proof stress for a stretch of 0.001 per cent, for annealed material of around 90,000 psi. tensile strength, ranges from around 10,000 psi. to 24,000 psi. in material of compositions varying around that usual for 18-8, while in the hard, cold-worked condition, at a tensile strength of around 250,000 psi., the 0.001 per cent proof stress is still 13,000 to 24,000 psi. The ability for very tiny plastic adjustment at relatively low loads is retained.

Too much cold work can damage a metal by "exhausting its ductility," and producing cracks, as is shown by "cuppy" wire with internal cracks due to overdrawing, and as has been discussed by Epstein (60) in relation to the layer severely deformed by punching. The way a metal responds to cold work is not told by ductility figures, but that does not mean that the tension test as a whole cannot give some of that information.

In studying the potentialities of the tension test, the damage line, the area within which no damage takes place under repeated stress even though the stress is above the endurance limit, and the area between the damage line and the *S-N* curve ought to be surveyed from the point of view of the plastic component of strain, at these stresses, which are low in comparison with the stress to failure.

Damping:

Hoyt (56) suggests using damping capacity figures determined at the stress of the endurance limit, divided by that stress, to give figures "having the dimension of strain." He recommends measurement, by some means, of plastic strain as a possibly helpful factor in studying the situation that exists at the stress level that counts in actual engineering use.

If damping data are examined as a criterion of ability for a trifling plastic flow at low stress, the conclusions are none too definite, perhaps because the usual methods of determining damping produce, not a uniform stress, but a stress gradient through the specimen, so that the test value is a resultant of all the stress levels. Torsional damping on thin-walled tubes might be much more enlightening.

Föppl (57) may have indicated the proper way to evaluate the qualities that differentiate toughness and brittleness. He makes calculations for soft steel, cast iron, and glass which seem to put them into the correct categories, though I have been unable to follow all his reasoning, especially that in regard to the utilization of damping values in the calculation, and therefore refrain from citing the figures. There are, however, enough instances, some of which I have cited above, to indicate that French, Föppl, and Hoyt are on a hot scent. Föppl's contention that the best steel would be one with no truly elastic range at all may be a sound one, even though the idea can, as yet, only be qualitatively expressed.

POSSIBLY IMPORTANT FACTORS

Bacon's (38) comparison of the behavior in notched endurance, cited earlier, is an indication that the absolute level of the yield strength needs to be brought into consideration.

Incidentally, if we come to the point where we must have a reliable evaluation of the ability to yield a trifle at a low stress for use as an aid in appraising crackless plasticity, we will probably use some more convenient method than the standard tension test: for example, temperature control will have to be used, as it is in a creep test, to eliminate inaccuracies from thermal expansion, and some standard number of cycles of repeated loading might be used to accumulate enough deformation for reliable measurement. Späth (59) has suggested apparatus for such a determination.

If the accumulated elastic deformation up to the primitive yield strength (that is, the stress producing some clearly defined, but very tiny amount of set) and the ratio of that deformation to the first increment of plastic deformation, reflect the degree of development of radial tensile stress rather than purely axial stress, at this crucial stage of deformation, it may be that Moore's "crackless plasticity" is a function of the modulus, the level of the primitive yield strength and the shape of the stress-strain curve over a very small plastic extension. Perhaps, in most engineering uses, what happens after that in regard to elongation and contraction is a matter of complete indifference. (Of course, if we happen to be using the plasticity of further deformation for formability in fabrication, or as insurance in case of accident, it is not a matter of indifference.)

Some of the puzzling lack of relationship between damping and endurance properties, and some of the peculiarities of notch sensitivity in relation to ductility, might be accounted for by failure to include the modulus and the level of the beginning of minute plastic flow in the comparisons. The metal may be trying to whisper, at the outset of plasticity, that is, through ductility *features*, just what we want to know, but we drown out that message by beating the bass drum of ductility *figures*. But since that assumption has still to be proved, we must admit that the ductility features of the tension test, as they have so far been used, have only limited significance.

I should be sorry if the suggestion that a precise determination of yield strength for very tiny set, modulus, and plastic strain in some early increment of plasticity, may be factors for making a qualitative appraisal of crackless plasticity, should set someone to concocting a specific quantitative formula, purporting to give a "merit figure of crackless plasticity." Factors peculiar to a given alloy can enter to disturb any such generalization. For example, even a slight plastic strain may "set off" a metallurgical reaction, such as aging, as French (49) pointed out, in which case the "stress-strain" diagram will picture the operation of something beside mere stress and strain. No matter how much we would like to untangle these Chinese puzzles along some prescribed formula, we should always keep in mind that computed figures may obscure rather than clarify the *features* of behavior that really count.

So many factors, favorable and unfavorable, may reinforce or oppose the ability for plastic deformation which we so roughly measure through the ductility figures of the tension test, that the significance of tensile ductility, by itself, should always be regarded as in doubt. It may be accompanied by the toughness we really want, or it may not.

In the absence of other information, tensile ductility figures serve as a rough, qualitative indication toward certain expectations, purely because of a background of past experience as to what other features have accompanied such figures. We shall all continue to use them in that fashion. But, as we continue to use them, can we not take steps to supplement or replace them with determinations that more directly and more truly measure the properties we really want?

As a first step, when we demand ductility because of the tacit assumption that it connotes something else, let us be honest and openly admit that we are making an assumption. Let us not put some figures from the tension test as we have been accustomed to make it, on a pedestal and worship them, but rather decide to study the tension test for significances that may lie hidden therein ready to show up if we will but look a little closer. Anybody can find a sun flower, but you have to hunt for the trailing arbutus.

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DISCUSSION ON SIGNIFICANCE OF THE TENSION TEST

MESSRS. D. J. MCADAM, JR.,[1] and R. W. MEBS[1] (*presented in written form*).—A tension test as usually made gives a stress-strain curve that does not represent the influence of stress on elastic strain. The indicated strain at any point in this curve is a combination of elastic with plastic strain. By a special procedure in the tension test, however, a distinction can be made between the elastic and plastic components of the strain at any stress. A single tension test thus gives correlated stress-strain and stress-set curves. From these may be derived several indices, which together give a fairly complete picture of the elastic properties and yield strength.

In such a test, the load is not increased continuously, as in an ordinary tension test. The test consists of a series of cycles of application and removal of load, with successive increases of the maximum load of the cycle. For each cycle the stress and strain are determined at maximum load, and the strain (permanent set) is determined after removal of the load. A stress-strain curve may then be obtained by plotting the stresses at the tops of the cycles against the corresponding strains, and a stress-set curve may be obtained by plotting the stresses at the tops of the cycles against the corresponding values of permanent set. By deducting values of permanent set from the corresponding values of total strain, a curve may be obtained to represent the influence of stress on elastic strain.

This correlation between stress-strain and stress-set curves is illustrated in the accompanying Fig. 1, which contains typical curves taken from a recent paper by the writers.[2] The curves in the lower row of this figure are typical stress-set curves obtained with cold-drawn 18 per cent chromium, 8 per cent nickel steel. Directly above each of these curves is the corresponding uncorrected stress-strain curve, represented by a broken line. From each of these pairs of correlated stress-strain and stress-set curves has been derived a (continuous-line) curve to represent the influence of stress on elastic strain.

The curves of each type in Fig. 1 consist of four pairs. The first curve of each pair was obtained after a short interval of tensile extension followed by release of load and by a relatively long rest interval. The second curve was obtained without intervening plastic extension, except the slight extension necessary for determining the first curve. The rest interval before determination of the second curve of a pair was much less than that before determination of the first curve. Comparison of the curves of a pair, therefore, gives information about the influence of duration of the rest interval, and of variation in the interval of prior plastic extension.

The ordinary stress-strain curve is not a sensitive representation of the relationship. The variation of strain with stress may be conveniently studied by means of stress-deviation curves, such as those in the upper row of Fig. 1. Abscissas of these curves represent deviations (strain differences) from an assumed initial modulus line. The nearer this assumed line is to tangency with the ordinary stress-strain curve at its origin, the more sensitive can the abscissa scale be made. The assumed values (E_A) of the modulus on which the initial modulus lines are based, are indicated in Fig. 1.

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[1] Metallurgist, and Junior Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

[2] D. J. McAdam, Jr., and R. W. Mebs, "Tensile Elastic Properties of 18:8 Chromium-Nickel Steel as Affected by Plastic Deformation," *Report No. 670*, Nat. Advisory Committee for Aeronautics (1939).

plastic and elastic components of the strain, at any point in an uncorrected stress-strain curve, is greatly influenced by a number of factors involved in the previous history of the metal. An incomplete view of the elastic properties of a metal, therefore, is obtained by considering either the stress-strain or the stress-set relationship alone. Consideration should be given to both relationships.

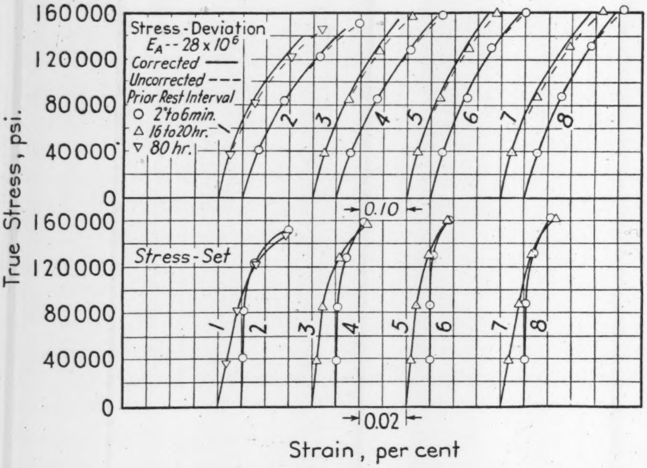

FIG. 1.—Correlated Stress-Deviation and Stress-Set Curves.

The first stress-set curve of a pair of this type, as illustrated in the lower row of Fig. 1, tends to be less steep than the second. The first stress-(elastic) strain curve of each pair, however, is initially steeper and more curved than the second. As illustrated by these comparisons, increase in the initial slope of the elastic stress-strain (or stress-deviation) curve tends to be accompanied by increase in the curvature, and by increase in the permanent set. As stated in the previously mentioned N.A.C.A. paper,[2] this relationship is not confined to 18 per cent chromium, 8 per cent nickel steel, but probably is a general relationship for metals. The ratio between the

As indices of the variation of permanent set with stress, the writers[3] have used "proof stresses" corresponding to five values of permanent set: namely, 0.001, 0.003, 0.01, 0.03, and 0.1 per cent. These indices are obtained from stress-set curves such as those in the lower row of Fig. 1. The 0.001 and 0.003 per cent proof stresses may be used as indices of elastic strength. The 0.01 per cent proof stress has been used in previous papers by one of us, as an index of yield strength. As shown in Table

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[3] D. J. McAdam, Jr., and R. W. Mebs, "The Tensile Elastic Properties of Typical Stainless Steels and Non-Ferrous Metals as Affected by Plastic Deformation and by Heat Treatment," *Report No. 696*, Nat. Advisory Committee for Aeronautics (1940).

III of a paper by McAdam and Clyne[4], the 0.01 per cent proof stress generally differs little from Johnson's limit. The 0.01 per cent proof stress evidently may be used as an index of yield strength. Other useful indices of yield strength are the 0.03 and 0.1 per cent proof stresses.

The elastic stress-deviation curves in the upper row of Fig. 1 are curved from the origin. Evidence has been accumulating for some time that such curvature is characteristic of the elastic stress-strain relationship for metals. The curvature and initial slope of the stress-strain line vary greatly with the metal and with its previous treatment. The curvature of this line and the variation of its slope with stress, may be conveniently represented by a method used by the writers in the previously mentioned paper.[2] This representation is illustrated in the accompanying Fig. 2. The graphs in this figure are derived from the elastic stress deviation curves in the upper row of Fig. 1. Ordinates in Fig. 2 represent stresses, and abscissas represent corresponding values of the secant modulus of elasticity.

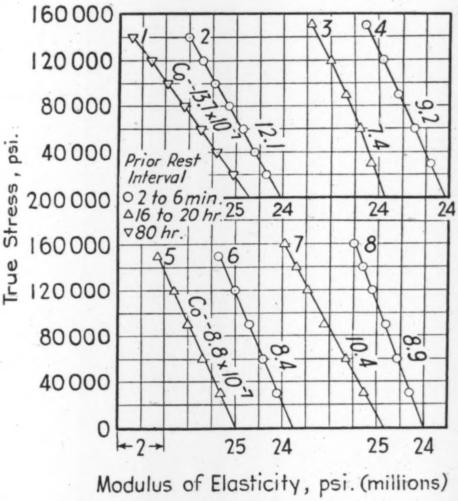

FIG. 2.—Stress-Modulus Curves.

The stress-modulus lines in Fig. 2 are based on the plotted points, which represent values derived from the corresponding stress-deviation curves. The stress-modulus relationship indicated by these lines is approximately linear. For annealed single-phase metals, the stress-modulus line generally is curved. Prior plastic deformation equivalent to more than about 5 to 15 per cent tensile extension, however, generally straightens the stress-modulus lines. Straight lines, therefore, generally are obtained with commercially cold-worked metals.

The value of E_0, the modulus at zero stress, is merely an extrapolated value, obtained by extending the stress-modulus line to zero ordinate. The linear variation of the modulus with stress may be represented by the equation $E = E_0(1 - C_0S)$. In this equation, E represents the modulus at the stress S, and C_0 is the linear stress coefficient of the modulus of elasticity.[5] This stress coefficient is proportional to the cotangent of the angle of slope of the stress-modulus line. When the stress-modulus line is straight, the corresponding stress-strain line is a quadratic parabola. When the stress-modulus line is curved, an additional term must be added to the equation. The equation then becomes

$$E = E_0(1 - C_0S - C' S^2).$$

In this equation, C' represents the curvature of the stress-modulus line.

When the stress-modulus line is straight, therefore, the course of the

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[4] D. J. McAdam, Jr., and R. W. Clyne, "Influence of Chemically and Mechanically Formed Notches on Fatigue of Metals," *Journal of Research*, Nat. Bureau Standards, Vol. 13, pp. 527–572 (1934).

[5] In the N.A.C.A. report previously mentioned, this index is designated C_1. In a second report, footnote 3, it is designated C_0. The index is expressed as fractional change of the modulus per pound per square inch.

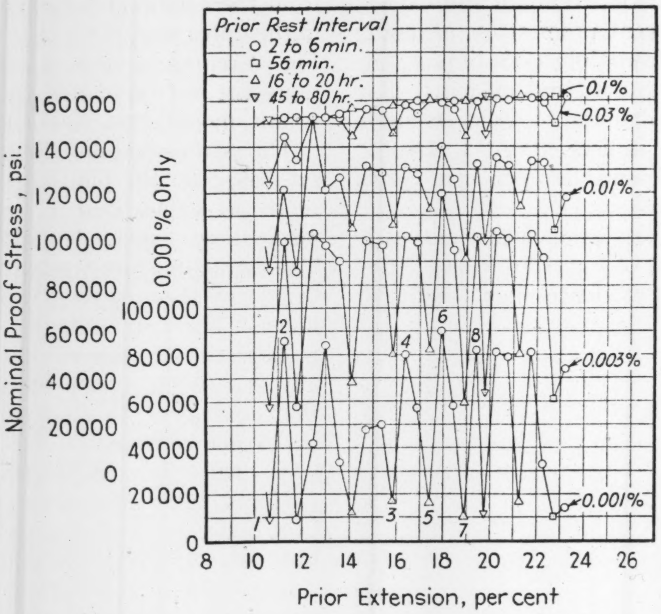

FIG. 3.—Variation of Proof Stresses with Prior Plastic Extension.

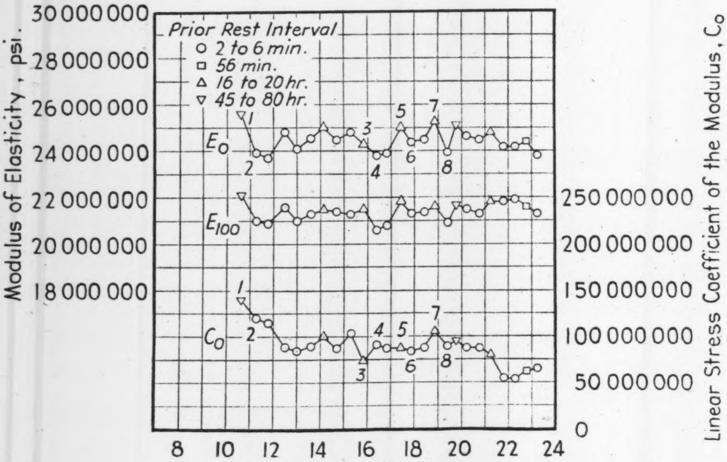

FIG. 4.—Variation of the Modulus of Elasticity, and of Its Stress Coefficient, with Prior Plastic Extension.

elastic stress-strain line is defined by the two indices, E_0 and C_0. These indices, however, give no information about the maximum stress that may be applied without excessive plastic deformation. This information is supplied by the corresponding values of proof stresses. By use of the two indices of the course of the elastic stress-strain curve, and the five previously mentioned indices of proof stress, a fairly complete picture may be obtained of the elastic strength of a metal.

The use of these seven indices in investigating the influence of various factors on elastic strength is illustrated by the incomplete diagrams in the accompanying Figs. 3 and 4. Both of these diagrams are based on data obtained by tensile extension of a single specimen of cold-drawn 18 per cent chromium, 8 per cent nickel steel. Figure 3 represents the variation of the five proof stresses with prior plastic extension; Fig. 4 represents the corresponding variations of E_0 and C_0. Four pairs of experimental points in each of these figures are derived from the curves in Figs. 1 and 2. The curves in Figs. 1 and 2 and the corresponding points in Figs. 3 and 4 are correspondingly numbered.

At these pairs of experimental points in Figs. 3 and 4 are abrupt oscillations of the curves due to the influence of the extension spacing and the duration of the rest interval. Such oscillations generally are qualitatively similar in the curves of Fig. 4. They are also qualitatively similar in the curves for the five proof stresses (Fig. 3). The oscillations in the proof stresses, however, are generally opposite in direction to the corresponding oscillations in the curves of variation of E_0 and C_0. This relationship is due to the previously mentioned tendency to opposite variations in the course of the stress-strain and stress-set curves, as illustrated in Fig. 1.

The use of this special procedure of tension testing is not advocated as a substitute for the shorter procedure in general use. The special tension test, however, may well be used in obtaining a more complete picture of the elastic strength and yield strength of any metal. It is especially useful for investigating the influence of composition and treatment on the elastic properties.

MR. W. R. OSGOOD[6] (*presented in written form.*—The three papers give an excellent summary of the present status of the tension test and of what may and may not be expected from it. The extensive bibliographies are especially to be commended.

It occurs to me to point out an essential difference between the trueness of Mr. McGregor's true stress and that of his true strain. His true stress may be so called because it represents a better approximation to the actual average stress on the cross-section than the stress as usually computed; but true strain seems to me to be a misnomer. P/A comes nearer representing an actual physical quantity than does P/A_0; but $\log (L/L_0)$ and $(L-L_0)/L_0$ are merely definitions of two different quantities, and to say one is true and the other fictitious has no meaning. It may be preferable, and there seems to be much to be said for it, to use the quantity $\log (L/L_0)$ where we have been using $(L-L_0)/L_0$, but that is no reflection on the latter quantity.

Is not Mr. Seely's condition *4* under which tensile-strength values are significant the same as or a special case of condition *3*? Mr. Seely states that in his discussion failure by elastic buckling is not considered. Possibly he meant to say inelastic buckling, for by far the most important property of a material in considering elastic buckling is the modulus of elasticity; and for most practical purposes this quantity, being the same or nearly the same in tension and compression, may be obtained as well from a tension test as from a compression test. When inelastic buckling may occur, however, the tension test is likely to have little or almost no significance except when the tensile and compressive yield strengths are definitely related to each other. The fact that most structural failures either are failures of compression members or are initiated by failures of a compression member would seem to indicate the importance of the compression test.

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[6] Materials Engineer, National Bureau of Standards, Washington, D. C.

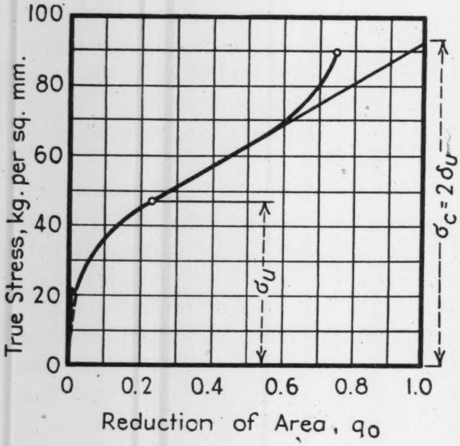

FIG. 5.—Relation Between True Stress and q_0 (Ludwik).

Mr. Gillett has covered the question of ductility most comprehensively. I should like to emphasize the importance of determining the elongation exclusive of that in the necked-down portion of the specimen. For estimating the degree of accident insurance present in a structural element under tension, it is necessary to know the elongation of the entire member at failure—only a short length of it will neck down. How much does the rest stretch? Most of the energy will be absorbed in this large rest. Although an actual structural member may not stretch the same amount outside of the necked-down portion as a test specimen of the same material outside its necked-down portion, nevertheless the elongation in per cent exclusive of that in the necked-down portion of a specimen should furnish a more accurate measure of the accident insurance than the elongation in per

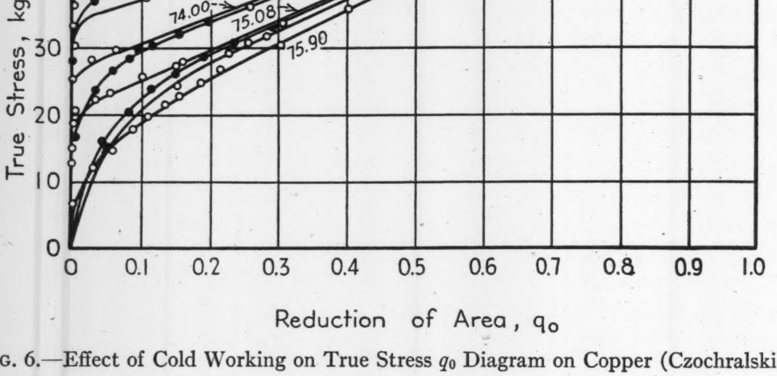

FIG. 6.—Effect of Cold Working on True Stress q_0 Diagram on Copper (Czochralski).

cent including that in the necked-down portion.

MR. R. E. PETERSON[7] (*by letter*).—In reference (**54**) of Mr. MacGregor's paper a diagram is given (accompanying Fig. 5) wherein true stress is plotted against q_0. Ludwik states that the "cohesive strength" (represented by the intersection of the extrapolated linear portion of the curve and $q_0 = 1$) is twice the true ultimate strength. He states further that there is a basic reason for this, referring to a paper published in 1913 which was not available to the writer. Perhaps the author is familiar with this work and could comment further. It is also evident that the data in Mr. MacGregor's Fig. 10 could be readily plotted in the same manner as Ludwik's data.

Ludwik refers to an article by Czochralski which contains an interesting result, shown in the accompanying Fig. 6. The various curves represent different degrees of cold working and all intersect at $q_0 = 1$. It is also to be noted that the actual true breaking strengths do not differ greatly. From these curves it would appear that if a relation such as Ludwik shows has any real basis, it would need to apply to a single state, such as a fully annealed condition. Mr. MacGregor's comments would be appreciated.

With regard to correlation of fatigue data, there should be a better chance of obtaining significant results working with true stresses and strains. We intend to obtain tension test data of this kind from material which we have used for fatigue tests. Since this is so easily done compared with obtaining fatigue data, it is suggested that this be carried out at as many fatigue laboratories as possible.

MR. H. H. LESTER.[8]—We at Watertown Arsenal regard the essential properties as yield strength and reduction of area. Yield strength is needed for design. The reduction of area gives a measure of the ductility.

I would like to ask Mr. MacGregor whether there is a definite relationship between the true breaking stress and the cohesive strength of the material.

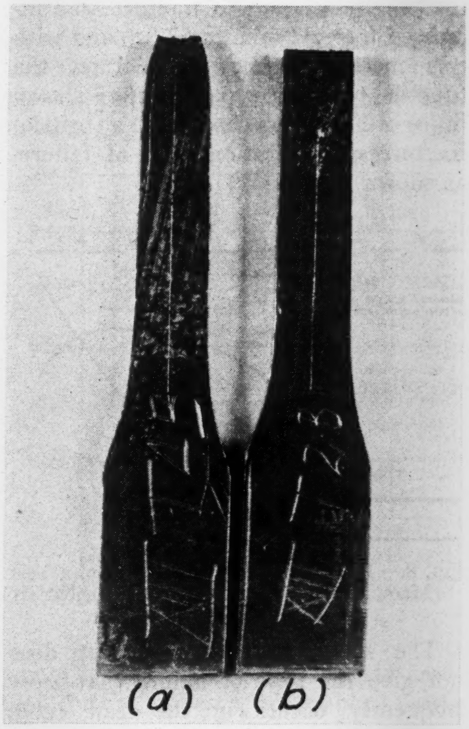

FIG. 7.—Showing the (Ductile) Yielding Type of Failure (*a*) and (Brittle) Fracture of Separation Type of Failure (*b*).

MR. C. W. DOLLINS[9] (*presented in written form*).—Mr. Seely points out that the time during which a metal is subjected to load is one of the conditions that may change the mode of failure from a (ductile) yielding type of failure to a (brittle) fracture or separation type of failure. This fact is strikingly shown by the failure of lead sheathing for electric cables. The accompanying Fig. 7 shows two specimens of the same material ($\frac{3}{4}$ per cent antimony-lead) cut from the same section of sheathing, and tested under steady load at room temperature. These specimens are as nearly identical as it is possible to make them. However by changing the steady tensile stress from 3000 psi. to 2000 psi., the length of time to fracture was increased from 32 min. to 42 hr., and with this increased time to fracture, the (ductile) yielding type of failure shown in (*a*) of Fig. 7 was changed to a (brittle) fracture or separation type of failure, as shown in (*b*).

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[7] Manager, Mechanics Division, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[8] Senior Physicist, Watertown Arsenal, Watertown, Mass.

[9] Engineer of Tests, Investigation of Lead Sheathing, University of Illinois, Urbana, Ill.

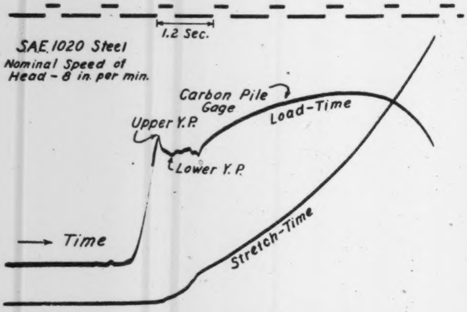

FIG. 8.—Typical Oscillograph Record of a Test Made Without the Use of the Weighbar.

The shorter-time tension test does not give the type of failure that most frequently occurs in the lead cable sheathing in service, whereas the much longer (fracture) test does bring out the type of failure most commonly met in service conditions. Thus, as Mr. Seely points out, unless the mode of failure in service is known, the significance of the strength features of the tension test is difficult to determine.

MR. PAUL G. JONES[10] (*presented in written form*).—Mr. Seely pointed out that the relation between upper and lower yield points of steel is dependent on the characteristics (especially springiness) of the testing machine. Rather striking evidence of this fact was indicated in recent tests made by the writer to determine the effect of rate of strain on the results of tension tests on metals.

The transition of mild steel from the elastic to the plastic stage appears to be a very sudden one causing a very abrupt change in the rate of loading at the yield point. As a result of this change, the inertia effects of the load-weighing mechanism used in the ordinary tension testing machine frequently produce considerable effect on the test results. In the tests here discussed, an attempt was made to reduce these inertia effects to a minimum. For this purpose, tests were made on standard $\frac{1}{2}$-in. machined specimens with shouldered ends in a relatively large and rigid (200,000-lb.) Riehle testing machine. Further, the load-weighing devices consisted of a compression block fitted with a carbon-pile telemeter for measuring lateral expansion under load. The change in resistance of the carbon pile with load was recorded by an oscillograph. Strains were measured by a resistance-wire type extensometer and recorded by the oscillograph.

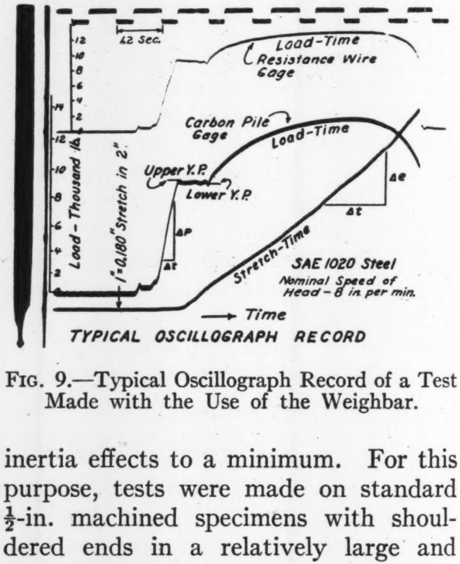

FIG. 9.—Typical Oscillograph Record of a Test Made with the Use of the Weighbar.

The accompanying Fig. 8 shows a typical oscillograph record of an S.A.E.

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[10] Instructor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

1020 steel tested at a nominal speed of head of 8.0 in. per min. On this record are recorded load-time and stretch-time relationships and a timing wave of impulses of 1.2 sec. It will be noted that with a load-weighing apparatus which has a small amount of inertia and springiness, the load-time curve indicates well-marked upper and lower yield points. Figure 8 also shows that the rate of loading and rate of strain in the yield point region are far from uniform. For the purpose of obtaining a check on the load indicated by the carbon-pile type of loading block, another load-measuring device was designed on a different principle. This device consisted of a 20-in. steel bar (weighbar) placed in series with the test specimen, having a reduced section over 10 in. of its length to which was attached a resistance-wire type of extensometer. A record of a test made on a specimen cut from the same bar and tested at the same head speed as that in the above test is shown in the accompanying Fig. 9. This record shows load-time curves obtained by both the resistance-wire gage and the carbon-pile gage and also a stretch-time curve. It is seen that there is very little drop in load at the upper yield point and that the rate of load and rate of strain in the yield point region is much more uniform than in the preceding test.

It seems evident that this difference in yield point phenomena may be attributed to the insertion of the weighbar in series with the specimen which decreased the rigidity or increased the "springiness" of the testing apparatus. It may be concluded that the behavior, at the yield point, of a structural member made of this mild steel would depend on the manner in which it is loaded, that is, on the rigidity of the adjoining members.

Tests were also made on the S.A.E. 1020 steel at nominal head speeds of 1.0, 0.4 and 0.05 in. per min. using the carbon-pile gage alone and with the carbon-pile gage in series with the weighbar. Results of these tests are shown in the accompanying Fig. 10 in which the yield point stress is plotted against the rate of strain in the elastic region. The results shown in Fig. 10 bring out the facts that the lower yield point is affected very little by the insertion of the weighbar; the effect of rate of strain on the lower yield point is much less than the effect on the upper yield point; and there is much less difference in the upper and lower yield points for the lowest speed of head used than for the highest speed of head used.

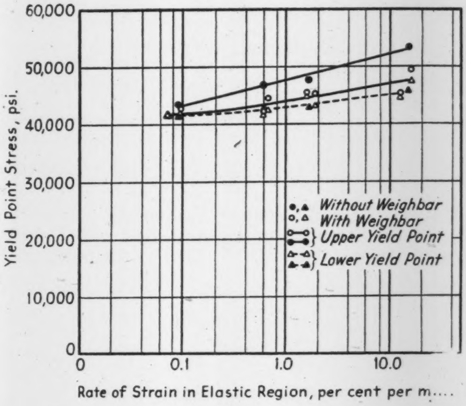

FIG. 10.—Effect of Weighbar on Yield Point of S.A.E. 1020 Steel.

MR. J. O. SMITH[11] (*presented in written form*).—Mr. Seely, on page 549 of his paper, states that in general for high yield ratio steels "in selecting design stresses for members of such material to be subjected to combined steady and variable stress, it may not be possible to take full advantage of the higher yield point of heat-treated high yield ratio steels, since failure by fracture (fatigue) may occur before the yield strength of the material is reached."

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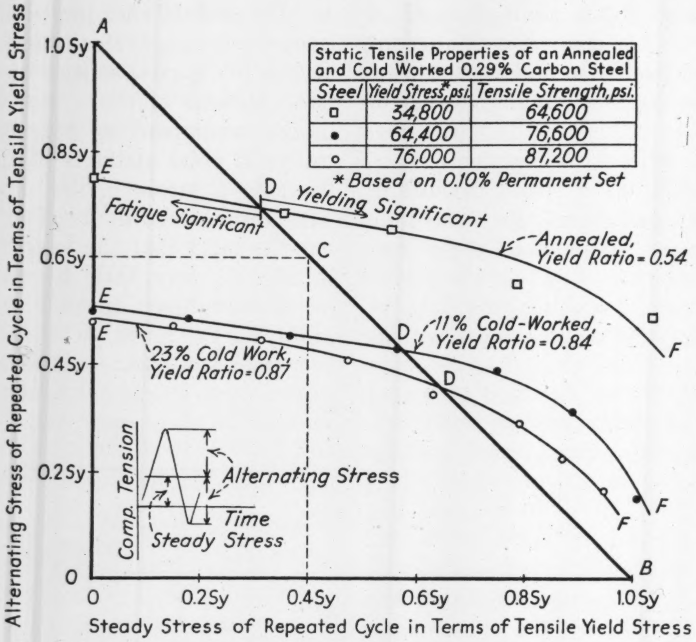
[11] Instructor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

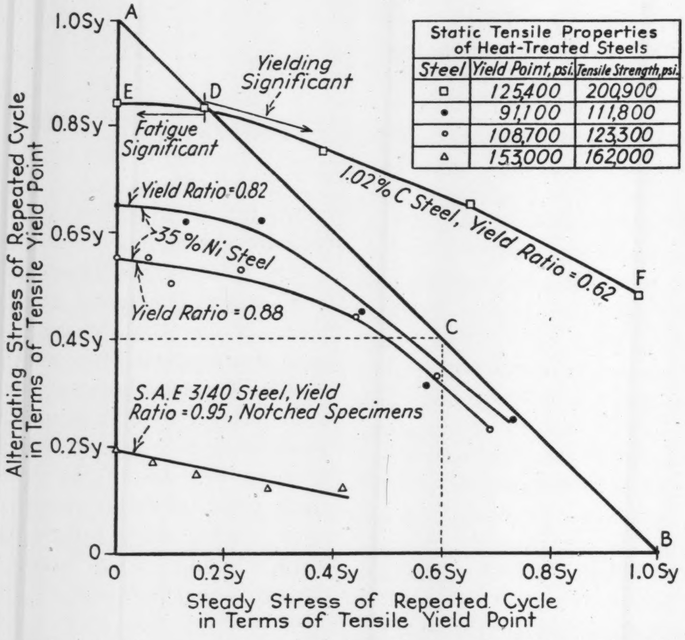


Static Tensile Properties of an Annealed and Cold Worked 0.29% Carbon Steel

Steel	Yield Stress*, psi.	Tensile Strength, psi.
□	34,800	64,600
●	64,400	76,600
○	76,000	87,200

* Based on 0.10% Permanent Set

FIG. 11.—Steady Stress - Alternating Stress Diagram Showing the Effect of Yield Ratio upon the Useful Strength of an Annealed and Cold-Worked 0.29 per cent Carbon Steel for Varying Ranges of Repeated Stress.



Static Tensile Properties of Heat-Treated Steels

Steel	Yield Point, psi.	Tensile Strength, psi.
□	125,400	200,900
●	91,100	111,800
○	108,700	123,300
△	153,000	162,000

FIG. 12.—Steady Stress - Alternating Stress Diagram Showing the Effect of Yield Ratio upon the Useful Strength of Several Heat-Treated Steels for Varying Ranges of Repeated Stress.

He bases his statement upon the consideration that the fatigue strength is not increased proportionately with the yield point when steels are heat treated or cold worked. This seems to imply that the strength features of the tension test are not significant for such steels used for resisting combined steady and alternating (variable) stresses. The writer feels that the significance of this implication has been somewhat neglected and wishes to discuss the statement quoted above by presenting data obtained by several investigators on both heat-treated and cold-worked steels having yield ratios which vary from medium to high values.

Moore, Jasper, and Howell[12] in reporting on several heat-treated steels having yield ratios ranging from 0.62 to 0.88 give data for the fatigue strength of polished specimens for various combinations of steady and alternating stresses in tension-compression. The writer[13] has reported data for a heat-treated steel having a yield ratio of 0.95 giving the fatigue strength of specimens notched by a transverse hole and tested under various combinations of steady and alternating torsional loads. In these torsional tests the steel always failed at the transverse hole by a tension (or helicoidal) fracture at 45 deg. to the longitudinal axis, so that the data might be regarded as representing the effect of a notch on the fatigue strength under tension-compression stresses consisting of cycles having various combinations of steady and alternating stress.

Brown[14] has reported fatigue strengths in tension-compression for a 0.29 per cent carbon steel which had been annealed and then cold worked by drawing through round dies and reducing the area 11 and 23 per cent, respectively. Thus, his results cover a steel which has a yield ratio in the annealed condition of 0.54, when cold worked 11 per cent of 0.84, and when cold worked 23 per cent of 0.87.

The data for both the heat-treated and the cold-worked steels are plotted on the accompanying steady stress - alternating stress diagrams (Figs. 11 and 12) which are similar to those used by Lessells in his paper in this symposium and also by Haigh.[15] In these diagrams the steady stress of a repeated cycle is plotted on the horizontal axis and the alternating stress is plotted on the vertical axis. See small diagram in accompanying Fig. 11; also see Fig. 1 of Lessells' paper in this symposium. Both steady and alternating stresses are expressed as fractional parts of the yield strength. Each of the plotted points in these diagrams represents an endurance limit for the material, that is, if the material is subjected to a steady stress equal to the abscissa of a plotted point, then the alternating stress represented by the ordinate of the point is the maximum stress which may be completely reversed about (superimposed upon) the steady stress a large number of times without causing a progressive (fatigue) fracture in the material. Thus, in the diagrams the plotted points which lie on the vertical axis represent endurance limits for complete reversals of stress, that is, the steady stress is zero and the alternating stress consists of cycles of equal tensile and compressive stresses.

The maximum stress in any cycle is equal to the sum of the steady stress and the alternating stress of the cycle. Thus if the line AB is drawn in the diagrams joining the ordinate and abscissa which are equal to the yield strength, then any point on the line AB represents a combination of steady and alternating stress which gives a cycle whose maximum stress is equal to the yield strength. For example, the point C on line AB has a steady stress of $0.6S_y$ and an alternating stress of $0.4S_y$ so that the sum of these is equal to S_y, the yield strength. Therefore, if one of the plotted points falls above the line AB, the sum of the steady stress and the alternating stress, that is, the maximum stress of the cycle, is greater than the yield strength, which would indicate that the specimen has failed by yielding before the fatigue limit (or fatigue strength) is reached. In this case the yield strength is significant as a basis for design stresses. If a plotted point falls below the line AB, then the sum of the steady stress and the alternating stress, that is, the maximum stress of the cycle, is less than the yield strength, and therefore the fatigue strength is significant as a basis for design stresses. If a plotted point falls on the line AB then the yield strength and fatigue strength are equally significant. The line AB will be referred to as the "yield line."

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[12] H. F. Moore, T. M. Jasper and F. M. Howell, "An Investigation of Fatigue of Metals," *Bulletin 136*, May, 1923, p. 67, and *Bulletin 142*, May, 1924, p. 72, Engineering Experiment Station, University of Illinois.

[13] J. O. Smith, "The Effect of Range of Stress on the Torsional Fatigue Strength of Steel," *Bulletin 316*, p. 18, Engineering Experiment Station, University of Illinois.

[14] R. M. Brown, *Journal of Engineering and S. in Scotland* (1928).

[15] B. P. Haigh, "The Relative Safety of Mild and High-Tensile Alloy Steels Under Alternating and Pulsating Stresses," *Proceedings*, Inst. Automobile Engrs., Vol. 24, pp. 320-347 (1929-1930).

Smooth curves are drawn through points representing fatigue strength data for various combinations of steady and alternating stresses for the given material. The points on this curve represent, therefore, the useful fatigue strength of the material. Points in the diagram above a curve represent a combination of steady and alternating stress which, when repeatedly applied, will cause a fatigue fracture. The curve for a given material will be referred to as the "fatigue curve" for the material.

It is now seen that the position of the plotted points in the diagram determines whether or not the danger of failure will be from fatigue (fracture) or from yielding. If the "fatigue curve" crosses the "yield line" (AB) the point of intersection marks the transition from fatigue significance to yield strength significance, that is, for example, for the 1.02 per cent carbon steel the "fatigue curve" intersects the "yield line" (AB) at point D where the steady stress is $0.16S_y$ and the alternating stress is $0.84S_y$. Then for combinations of steady and alternating stresses represented by points on the left-hand branch ED of the curve, that is, to the left of point D, the fatigue strength is significant; but, for combinations of steady and alternating stress represented by points on the right-hand branch of the curve DF the yield strength is exceeded by the maximum stress of the cycle before the fatigue strength is reached, so that the points along the "yield line" included between D and B represent the limiting combinations of steady stress and alternating stress which can be applied to the material without causing a yielding failure.

Examination of the diagrams for the heat-treated steels shows that if the yield ratio is low, the transition from fatigue significance to yield strength significance takes place at a relatively low steady stress, indicating that the yield strength is the maximum useful strength for this material for a rather wide range of steady stress-alternating stress combinations, and hence the tension test gives significant values for these combinations. For such a material the yield strength is therefore very significant when selecting design stresses for repeated stress conditions. On the other hand, if the yield ratio is high, or if stress concentrations are present, the fatigue curves may not intersect the yield line (AB) so that for all combinations of steady stress and alternating stress the fatigue strength is significant, that is, the yield strength is not significant for design since the material does not fail by yielding and hence full use may not be made of the high yield strength.

The diagrams for the annealed and cold-worked steel show that the higher the yield ratio becomes by cold working the material, the wider the range of significance of fatigue becomes, that is, the point of transition *D* from fatigue significance to yield significance moves further to the right increasing the length of *ED* and decreasing the length *DB*. This means that for high yield ratio steels produced by cold working, the role of significance of the yield strength is reduced so that full use of the high yield strength may not be used for the stress combinations discussed here.

MR. G. C. RIEGEL[16] (*presented in written form*).—Mr. Seely has given us a most interesting and readable discussion of the significance and limitations of the common tension test. Thousands of dollars are wasted annually by makers and consumers of steel in conducting tension tests which, in so far as predicting the serviceability of the material tested, are worthless criteria.

As has been pointed out by others, scarcely any failures in engineering materials come from purely tensile stresses. The actual mechanism of failure is usually entirely of a different kind.

In good design, usually the factors of precisely determined elastic moduli, of rigidity, and of safety, have been observed. As a consequence, most of the failures of engineered parts come from other causes, the most disastrous of which are the sudden or brittle type of separation, and the progressive type. Too frequently both types occur without adequate warning.

An axle shaft can have failed by progression until less than 10 per cent of the section is intact, when suddenly it twists in two. A tie rod of tough steel may have failed by progression from a thread notch until but a "waning moon" segment of the section remains, when suddenly it separates with a brittle fracture.

A drive gear may perform satisfactorily for years until, with the consequence of wear and power train "windup," so much stress can be applied on one tooth that it suddenly parts from an otherwise tough material body, as measured by either tension or notched-bar impact tests.

A large box section made by welding plates to channels and formed into an arch can fail by brittle and sudden separation throughout the section when suddenly dropped less than 2 ft. in cold weather. There may be practically no evidence of plastic deformation prior to, or at the point of, failure.

From one such example, tension tests and other tests were taken adjacent to the fracture. The results follow:

Tension test taken transversely to the direction of rolling, tested at room temperature (75 F.):

	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Contraction of Area, per cent
75 F. . .	56 200	33 800	45.0	50.0
−20 F. . .	59 300	33 800	42.0	53.8

Bend tests made at both +75 F. and −20 F. gave 180-deg. angle of bend without failure.

Notched Bar Impact Tests (Charpy Type)

	Single Width, ft-lb.	Double Width, ft-lb.	Triple Width, ft-lb.
T[a] at +75 F. .	25.0	50.0	71.0
P[a] at +75 F. .	36.0	84.0	98.0
T at −20 F. .	2.75	4.25	7.25
P at −20 F. .	2.00	3.50	7.50
T at −40 F. .	1.25	3.00	5.00
P at −40 F. .	1.25	4.00	6.25

[a] T = Transverse. P = Parallel to the direction of rolling.

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[16] Chief Metallurgist, Caterpillar Tractor Co., Peoria, Ill.

Chemical analysis of the above material:

Carbon, per cent	0.16
Manganese, per cent	0.46
Silicon, per cent	0.04
Nickel, per cent	0.09
Chromium, per cent	0.03

An example of a torsion failure could also be cited which could hardly be evaluated in terms of tension. A pinion made of S.A.E. 2345 steel and hardened to Rockwell "C" scale hardness of 52 to 56, had continued to fail suddenly under torsional stress, whereas a pinion of the same material when reduced to a Rockwell "C" scale hardness of 34 to 37, would show sufficient "wind-up" and release to withstand the shock load in torsion.

It is pleasing, therefore, to see that Mr. Seely has painted the whole picture with a "broad brush" since many of the tests which have been performed in a laboratory method are poorly calculated to correlate with the conditions encountered in service. To continue to test material in tension and fail to take into account other more simple and equally reliable tests whose correspondence to the mechanism of incipient failure would yield greater value, both as a gage for maintenance of minimum standards of quality, as well as the diagnosis of parts which have failed under the usual conditions of stress, gives the appearance of engineering fixity in a world of progression.

MR. BRUCE JOHNSTON[17] (*presented in written form*).—Mr. Seely has explained clearly two basic modes of failure which may take place in a metal which behaves in a ductile manner when stressed in uniaxial tension. These are the yielding, associated with shear stresses, and the cohesion failure associated with triaxial tensile stresses. Many structural elements built up of relatively thin plate material are stressed principally in a single plane, or biaxially, whereas in heavier machine elements, castings, built-up welded units, or a bar sharply notched on all sides of a particular section there may be appreciable triaxial stresses. Initial yielding of metals has been explored experimentally for the various biaxial states of stress, in which cases the shear stresses are always equal to or greater than in a simple tension test. Metals which have either a gradual or drop of the beam yield in uniaxial tension have been found to agree with the shear-strain-energy theory of yielding for biaxial stress combinations. Nadai[18] has shown that this theory may be expressed exactly without recourse to strain energy criteria by the following statement: "Yielding will result when the octahedral shear stress reaches a certain limiting value." This limiting value may most easily be determined by the simple tension test, in which case the octahedral shear stress is equal to 0.47σ, where σ is the unit tensile stress. The octahedral shear stress may be defined as the shear stress which always exists simultaneously and in exactly equal magnitude on the eight planes whose normals make equal angles with the principal stress directions. It is numerically equal to $\frac{1}{3}\sqrt{(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2}$ where σ_1, σ_2, and σ_3 are the principal stresses. The simple tension test is thus important in establishing a fundamental constant in the criteria for yielding.

Triaxial tension is probably a relatively rare state of stress in most structural elements. The strength theories have had little experimental verification in the triaxial state, due principally to experimental difficulties. Assuming the correctness of the octahedral shear stress theory, however, the following conclusions are reached. If the principal triaxial tensile stresses are equal in magnitude, the octahedral shear stress is zero and the failure must be cohesive failure. If the principal triaxial stresses are not equal, and increase in constant ratio, the critical octahedral shear stress will eventually be reached, provided of course that prior cohesive failure does not take place. Seely, Hoyt, and others have pointed out the desirability of knowing the "statistical" cohesive strength of the material. This is certainly an important constant and is not determined by the simple tension test for the unworked, ductile material.

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[17] Assistant Director, Fritz Engineering Laboratory, Lehigh University, Bethlehem, Pa.

[18] A. Nadai, "Theories of Strength," *Journal of Applied Mechanics*, Vol. 1, No. 3, July, 1933, p. 111.

The work of Kuntze[19] in attempting to evaluate the cohesive strength by use of cylindrical, V-notched specimens needs further study, corroboration, and extension. Neuber[20] has shown by the theory of elasticity the triaxial state of tensile stress which exists in deep and relatively sharp hyperbolic notches. A particular case of one of Neuber's general solutions is shown in the accompanying Fig. 13. It may be seen that very nearly equal triaxial tensile stresses exist in a relatively large area at the minimum cross-section of the notch.

Mr. Seely has indicated in Table I a general outline of tests to show more completely the strength features of a material. The Fritz Laboratory of Lehigh University already has embarked on tests of a similar nature. Tests on different structural metal alloys are to be made covering most of those in this table but restricted to zero previous deformation and normal temperature. Mr. Seely points out the additional need of tests which will reveal significant structural behavior. To a limited degree such tests are also included in this program. The same alloys tested physically are to be tested for structural behavior in various types of joints. Machining of the specimens is in progress but it will be more than a year before the present program can be completed. The work is being done in collaboration with the A.S.C.E. Structural Division, Committee on Design of Structural Members. Mr. Seely also discusses the important question of the ratio between yield point and ultimate strength. In view of arbitrary clauses which limit this ratio in some specifications it is hoped that the Lehigh tests, as well as others of a similar type now in progress elsewhere, will be revealing.

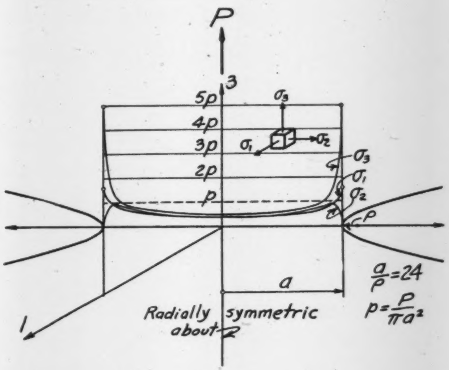

FIG. 13.—Cross-Section Showing Triaxial Stress Distribution in Deep Hyperbolic Notch from H. Neuber.[20]

MR. W. LEIGHTON COLLINS[21] (*presented in written form*).—Under the heading of Yield Strength, Mr. Seely states that some engineers doubt whether a metal can undergo sufficient plastic flow to make the adjustments required for the successful functioning of machine and structural members unless the metal has a well-defined yield point in the tension test.

In attempting to compare various ductile steels (including the five steels whose stress-strain diagrams are shown in Fig. 1 of Seely's paper) on the basis of their ability to make adjustments to localized stresses, under static loads, by yielding without evidence of cracking, the writer has used a tension specimen containing a short turned-down portion—a square-shouldered notch. The dimensions of the specimen and the notch are shown in the accompanying Fig. 14. Other types of notches have also been used, but this one illustration serves the purpose of this discussion.

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[19] W. Kuntze, "Kohasionfestigkeit," *Mitteilungen* der Deutschen Materialprüfungsanstalten, Sonderheft XX (1932).

[20] H. Neuber, "Kerbspannungslehre," Julius Springer, Berlin (1937).

[21] Assistant Professor, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

The typical "stress-strain" curve for one steel tested is also shown in Fig. 14. A 2-in. gage length, including the notch, was used; all stresses, except as noted, are based on the area of the reduced section; and the strains are average values for the 2-in. gage length.

Examination of the curve reveals that there are two stresses at which marked yielding took place, that is, two stresses at which deformation continued without an increase in load. It is felt that the first yielding, at *A* in Fig. 14, started as a localized yielding at the fillets, which relieved the stress concentration, produced a more uniform distribution of stress, and permitted sufficient general yielding throughout the turned-down length to indicate a yield point. The strain hardening accompanying this yielding enabled the load to be increased until the main body of the specimen near the notch yielded, thus indicating a second yield point, *B*, in Fig. 14. The unit stress, based on the large area, at which this second yielding occurred is nearly the same as the yield point of the material as determined for unnotched specimens. Additional evidence of the ability of the material to make local adjustments at the abrupt change in section is borne out by the increase in strength beyond the second yield point.

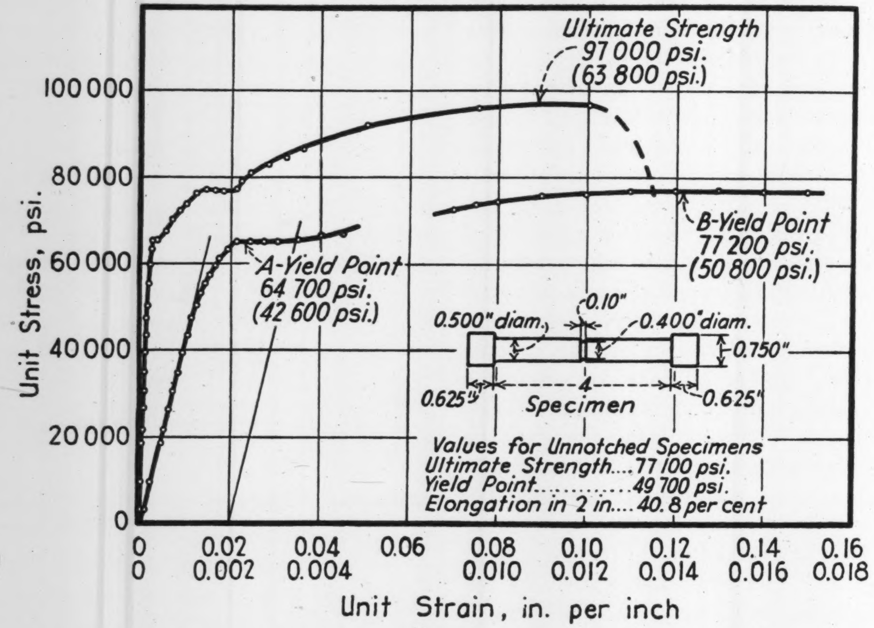
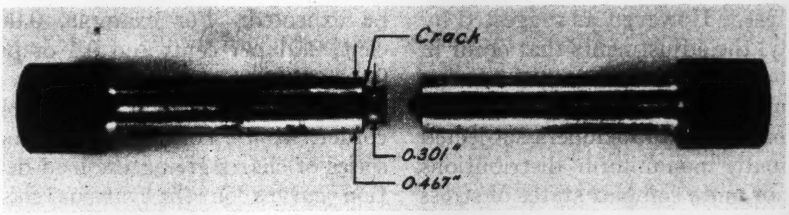
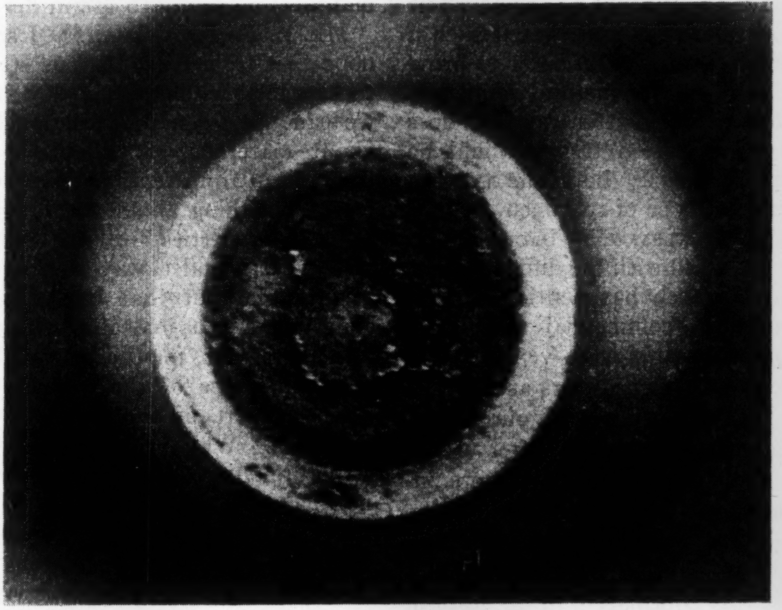


FIG. 14.—Stress-Strain Diagram for a Tension Specimen Having a Rectangular Notch. Values in parentheses are based on 0.500-in. diameter.

Visual evidence of the actions described (ability to yield plastically without cracking and relief of localized stress) can be obtained from an examination of the ruptured specimen shown in the accompanying Fig. 15 (*a*). The necking down of the turned-down length (reduction of area equals 45.0 per cent) is readily observable and the dimensions given indicate the measurable yielding (12.8 per cent reduction of area) in the main body of the specimen. Also visible in the figure is the crack which opened up at one of the fillets when the maximum load was reached; the other crack obviously spread and led to complete fracture of the specimen.



(*a*) Longitudinal view.



(*b*) Cross-sectional area (× 4.6).

FIG. 15.—Ruptured Specimen.

The appearance and shape of the area on which the specimen ruptured indicates the complex state of stress (undoubtedly three-dimensional) existing at the notch. The inward progress of the crack is marked by the smooth, rather shiny area marked *A* in Fig. 15 (*b*). The central portion of the cross-section, marked *B* in the same figure, is coarse and granular in appearance and is the part that fractured suddenly. Such differences in the appearance of the area of fracture are also found when a V-notch is used. However, the cracks do not spread equal amounts for all steels; in fact, one steel failed completely and suddenly without the formation of a crack.

Although the experimental work so far done has not shown conclusively whether the steels used can be sharply differentiated on the basis of the test just described, some differences have been observed and it is felt that evidence of two "yield points" in a specimen having a specified short notch may be more convincing evidence of the ability to make structural adjustments than a well-defined yield point in the standard tension test. However, as suggested by Mr. Seely, the adjustments that occur in a specimen, even in a notched tension specimen, may not simulate the adjustments that occur in members subjected to nominally nonuniform distributions of stress or more complex states of stress than exist in a tension specimen. It is also recognized that different notches would have an influence on the behavior of various materials and that if such a test were standardized in order to bring out the significant structural properties of the material in a better way than does the standard tension test, it would be necessary to specify both the type of notch and the type of specimen.

MR. O. C. RUTLEDGE[22].—In our laboratory we are faced with the difficult situation that Mr. Seely has mentioned. We have to make commercial acceptance tests that will satisfy the vendors, and ourselves and our customers as the purchasers. On the other hand, our designing engineers are always after us to furnish them with more and more information on the properties of the materials that they want to consider for use in their designs. We usually make up this information in the form of a chart or table, listing as many of the common properties as are available and continually adding to them as more information is collected. On the materials that show a yield point, the physical properties listed would include proportional limit, yield point, tensile strength, elongation, and reduction of area, and fatigue endurance limits, and a general discussion of the class of applications that the material might well be used for. On materials which do not show a definite yield point, we have had under consideration listing a table of stresses at which various amounts of offset might be expected. For example, 0.001 per cent, 0.01 per cent, and 0.1 or possibly 0.2 per cent, and more recently, since we have purchased an autographic recorder, we are considering making up a series of charts giving the load-deformation curves on the various classes of materials so that the engineer can more ably choose for himself what particular materials he wants to use in his design.

MR. L. B. TUCKERMAN.[23]—I sincerely hope that from these various studies of tension tests we shall some day find that we can use them to learn much more about materials. It would be highly desirable if from tension tests we could learn something about the fatigue and impact properties of materials. We have yet to find a way to do that. I might point out that the principle of similitude fails us in dealing with crystalline aggregates, for the reason that the size and shape of the crystals are an essential part of the behavior of the structure. When you make a smaller structure out of the same metal, you do not make the crystals smaller in proportion and consequently there is a severe limitation on the application of the principle of similitude. It is known that in the Charpy impact test you cannot, on the basis of dimensional reasoning, correlate a test on a large specimen with a test on a smaller one.

These comments give, perhaps, too pessimistic a view of the tension test. In a large number of structures, a relatively large portion of the material is stressed in practically pure tension. We know from many tension tests that if the material fails in the tension test at stresses below the tensile stresses in the structure, the structure will not stand up in service. Consequently we must know the result of tension tests on the material. That is basic. We must have it.

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[22] Member of Engineering Staff, Works Laboratory, General Electric Co., Schenectady, N. Y.

[23] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

We need also to know the compressive properties of the material. Compression tests are time consuming and costly. It is to be hoped that some day we can find a definite correlation between the tensile properties of materials and their compressive properties. One of the most definite things we know about the strength of structures is that the shape of the compressive stress-strain curve either at the yield point, if the material has a yield point, or in the neighborhood of the yield strength (I hate those words, I voted against them), is a criterion upon which we can rely, and rely fully, in determining the nature of the transition between critical elastic instability and plastic instability in structures stressed partly or wholly in compression. Failure by elastic or plastic instability is characteristic of practically all aircraft structures, in particular, stressed skin structures.

I think there is too much of a pessimistic tone in Mr. Gillett's paper.

If you take samples of a uniform material and subject them to a series of processes, uniform except for one variable parameter, it is a reasonable expectation (and experience gives a reasonable confirmation to that expectation) that there should be a close correlation between any two mechanical tests made on those samples. Thus if you take samples of a given steel such as a S.A.E. 1040, quench them at a given temperature, and draw them at a series of different temperatures, you expect, and within reasonable limits, will find, close correlation between the resultant tensile strength, elongation, reduction of area, Brinell number, etc.

If you take samples of another kind of steel such as a S.A.E. 4140 and put them through the same processes, you expect and, within reasonable limits, will find, close correlation between the same resultant physical properties.

However, the relationships between these properties for the two steels will not in general be the same. For example, S.A.E. 4140 will have a much greater elongation corresponding to high Brinell numbers than will S.A.E. 1040 and the curve of change of elongation with Brinell number will have a different shape.

In fact it would be sheer accident if they were the same except in the improbable, but conceivable, case that two superficially different tests measured in different ways exactly the same properties of the materials.

The steels covered by either A.S.T.M. Standard Specifications A 7 – 39, or A 212 – 39[24] do not, of course, form a one-parameter system in the sense stated above but they are sufficiently nearly one parameter systems that there is for them a fair negative correlation between tensile strength and elongation which is recognized in the specifications by specifying a minimum elongation given by a ratio "constant to tensile strength."

The trend noted by Templin (p. 568) is another illustration of a rough, in fact, very rough, approach to a one-parameter system, and Lundgren's breaking steels into classes (p. 568) is another stab at one-parameter systems.

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[24] Standard Specifications for Steel for Bridges and Buildings (A 7 – 39) and for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 4½ in. and Under in Thickness) (A 212 – 39), 1939 Book of A.S.T.M. Standards, Part I, pp. 1 and 60, respectively.

Russell's report (p. 569) of no change in notched endurance with lowered elongation is not in conflict with this. He had a close approximation to a one-parameter system and the correlation coefficient of a horizontal line can be as high as that of a line with any slope.

I think that examination of all the cases in which correlations are reliably reported between impact values and polished or notched endurance values on the one hand and tensile elongation or reduction of area on the other, will show that the correlation is greater the more nearly the specimens form a one parameter series.

I hold no brief for the specific values of elongation specified in A.S.T.M. specifications for structural steel and certainly not for the slight differences noted, but I do not like the assumption that no real justification exists for them. A real justification *does* exist for specifying some minimum of elongation for many structural materials and it lies, in the fact, learned from experience, that, for some classes of material, elongations below a certain value are almost sure indications of unsuitable material. If a structural steel was supposed to conform to A 7 – 39 and in the usual tension test it showed only 5 per cent elongation you would know that something was wrong with it. That is, of course, extreme, but I have tested materials submitted under similar specifications which gave just as extreme values and I did not feel it necessary to question whether the specified elongation was "actually needed" before rejecting the material.

Two cases cited are excellent illustrations of this. Sims and Dahle's "hair trigger difference in ductility values" apparently did exclude some bad cast steels.

Further, regardless of whether "the engineering requirements" of the Mount Hope Bridge wire "actually needed" the 30 per cent contraction specified, the occurrence of sporadic specimens with much lower contraction should have (hindsight is better than foresight) warned the engineers that there was "something rotten in Denmark."

I am firmly convinced that it *is* sound specification writing, for a given class of materials, "to demand, without qualification, a definite figure" of tensile elongation "without knowing that the engineering requirements actually need that figure" provided it "without *excessive* cost, can be obtained without *excessive* sacrifice of other desirable properties."

Manufacturers will complain if the costs are excessive, and users will complain if other desirable properties are excessively sacrificed and specifications can always be revised where it is proven desirable.

Such specifications will, of course, unavoidably cause the rejection of some perfectly serviceable materials for failing to meet an arbitrary figure. If the failure to meet the specifications is caused by poor mill control, they should be rejected as the same poor mill control could as readily produce dangerous material and the elongation furnishes, for many metals at least, a criterion which surely excludes much poor material.

The definite figure which has proved satisfactory in selecting one class of material for a given use, is of course not sacrosanct, and has no bearing whatever on the figure to be set for "a different material, really superior" for the same use. The only real hope I can see for "potential users" and also for specification writers who think that it is sacrosanct, is to educate them out of their ignorance.

When, for example Grotts can produce a perfectly satisfactory alloy steel casting with 44 per cent contraction, while the specification calls for a minimum of 45 per cent, it is perfectly in order to urge a lowering of the requirements for reduction of area in A.S.T.M. Standard Specifications A 87 – 36[25] *provided* that there is specified some other adequate safeguard against the occurrence of "the film type of inclusion," which I presume to be seriously detrimental.

However, if I were urging the lowering of this requirement I would also urge that it be lowered *no more than enough* to include known demonstrably satisfactory cast steels, and before I urged even so much, I would want to be perfectly sure that there was some other adequate safeguard against "the film type of inclusion" or other seriously detrimental defect.

I am reminded of a talk I gave 7 yr. ago. It does not cover all the points but it seems to me sufficiently pertinent to quote:

"So far, I have mentioned only the results of the better understood materials tests in judging this relatively new material. What of the others? One example will have to suffice. For the usual high-grade structural duralumin an extension of 18 per cent in 2 in. is specified. Why? For some of the new modifications extensions as low as 12 per cent are considered satisfactory. Why? We specify about 25 per cent for ordinary structural steel because we feel that smaller elongations indicate material less secure against shock and fatigue failure as well as more difficult to fabricate, and 25 per cent is readily obtained commercially. It is hardly safe to reason by analogy from steel, for we have to do with an entirely different material. This is clearly shown by the age-hardening properties of duralumin, which make the practical difficulties of its fabrication so different from those of steel. By lowering the specified elongation of duralumin to 12 per cent, material is now furnished for special purposes up to 75,000 psi. tensile strength instead of 55,000 to 65,000 psi. which was formerly considered the best that could be furnished commercially.

"These newer, higher-strength alloys with lower elongation, developed to meet specific demands, introduced and used with many misgivings, have so far proved to be satisfactory in service. It is still too soon to predict the ultimate judgment which will be passed on them.

"Could elongations even lower than 12 per cent be specified with safety? Who can say?

"A new material, however desirable its qualities, must thus pass through a period of probation before designs are worked out which will use these properties to the best advantage. In this probationary period structural tests furnish one of the most valuable aids to rapid progress. Even materials which have been much longer known and used, have as yet not wholly passed this probationary period, so that structural tests on so well known a material as ordinary structural steel are still teaching us how it may be used to better advantage."

THE CHAIRMAN (*Mr. J. M. Lessells*[26]).—The paper by Mr. Gillett refers to the Ford cast crankshaft. While I have been associated for several years with questions relative to the suitability of forged alloy steels for aircraft engine crankshafts, the question of the use of cast material for such parts cannot, on the basis of the success which has been obtained with the cast automobile crankshaft, be disposed of on the grounds of a decrease in the ductility values alone. Mr. Gillett in his paper has put this situation before us in a rather clear manner. Other factors may, however, limit the use of cast materials for crankshafts.

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[25] Standard Specifications for Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 – 36), 1939 Book of A.S.T.M. Standards, p. 224.

[26] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

MR. T. MCLEAN JASPER[27] (*presented in written form*).—I like the very tone of Mr. Gillett's paper. I hope that the paper will act as a "Stop, Look and Listen" sign for a group of honest, hard-working but sometimes headstrong testing engineers and specification writers. We truly sometimes strain at little gnats and swallow whole strings of large camels.

The writer would introduce yet another thought into the picture that ductility may play in a significant manner in producing final engineering structures. There are certain structures which depend upon the yield point to determine the load at which they will fail. This applies particularly to the problem of collapse or buckling such as is found in tubes used for deep oil wells. The stress-strain diagram in compression for certain steels is very different from that in tension and steel may be compressed under restraint so that its compression yield is very materially raised in the most useful direction. In compressing a tube a certain amount of the measured original ductility is utilized and it is significant that a nonductile material will not yield to this type of treatment without fracture.

It is possible to raise the compressive yield strength of certain steels above the ultimate strength in tension in the same direction without utilizing more than about one-quarter of the available ductility, thereby allowing three-quarters of the original ductility to take care of the hazards of operating the casing. This three-quarters of the original ductility as measured by elongation may be more than the total ductility representing the normal steels used in the several casing problems.

At a recent meeting of the American Petroleum Institute the writer proposed that structures be given study so that their fundamental ductility, or perhaps toughness, requirements might be arrived at. This is a problem worthy of the American Society of Testing Materials. As Mr. Gillett has pointed out in general, as strength is increased ductility decreases. A steel heat treated for high strength is invariably less ductile in this state than if in the annealed or normalized state. The Society has been giving careful study to impact testing for a number of years. I would propose that a committee be set up to study the significance of various forms of ductility measurements in its application to engineering requirements. It may well be that such a study will result in decreasing its importance in specifications.

MR. S. W. LYON[28] (*presented in written form*).—Mr. Gillett's discussion merits the careful consideration of all engineers having to do with the testing and use of engineering materials. It is not so much for the data given in this paper but rather for the comprehensive gathering together of the ideas relating to the numerous phases of a subject much in need of consideration, that makes this paper of high merit. This paper should stimulate several lines of thought and endeavor which will ultimately bring understanding to a subject much in need of clarification.

A consideration of the service requirements of railroad rails and the ductility figures of rail steel as given by the tension test offers a good example of how a material with rather limited ductility fulfills the demands of severe service in a satisfactory manner. Rail steel, a tonnage product, with an average carbon content of 0.75 per cent, is usually considered as a brittle material as shown by elongation values averaging 9 or 10 per cent as obtained from the usual tension test. Yet rails in track even in the best of maintained main line are by no means rigid, for some vertical movement is always present under load. Rails are also called upon to support high repeated moving loads, and these loads at the gaps existing at rail joints further subject the rails to impact. On rare occasions the dynamic augment from an accidentally out-of-balance locomotive operated at high speed has imposed impact loads of great enough magnitude to kink the rails permanently, but fracture seldom resulted directly from such cause. Further, rails give service under rather wide ranges of temperature conditions, certainly under corrosive atmospheric conditions, and with the presence of occasional casual stress raisers such as nicks imposed during operation and maintenance. It seems safe to say that relatively few rail failures can be traced to insufficient ductility of the steel.

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[27] Director of Research, A. O. Smith Corp., Milwaukee, Wis.

[28] Talbot Laboratory, University of Illinois, Urbana, Ill.

With all this, if rails can give good service under severe service conditions even though the rail steel is of relatively low ductility, is it necessary to require such high ductility figures for materials operating under service which impose much less severe requirements from a service standpoint?

MR. BRUCE JOHNSTON[29] (*by letter*).—Mr. Gillett's paper focuses attention on the need for more accurate knowledge regarding ductility requirements for a particular structural or mechanical use of a particular material. He cites numerous instances where the engineer gets along without a high degree of plasticity. The crankshaft he describes is an excellent example. Although Mr. Gillett has indicated briefly the ductility requirements in the structural field, the need for fairly large plastic extension is not so rare as might be inferred.

In the steel bridge or building plasticity of material is needed in each of the three stages of (1) fabrication, (2) erection, and (3) service. In fabrication, the cost and methods used in punching, shearing, and bending operations certainly depend to a large degree on the plastic deformability of the metal. This plastic deformability is at least partly indicated by the ductility features of the tension test. In the erection stage, punched holes in a riveted structure may not line up, and must be drifted into line. In very local regions the resulting plastic dislocation may be considerable. After the bridge or building is in service the actual structural elements may have local uncalculated stresses due to applied load which may locally cause plastic flow.

The problem is not simply one of local redistribution of stress in one particular part which would require probably less than 1 per cent total strain. For example, a riveted joint is designed on the basis of assumed equal load distribution among the rivets. In a long row of rivets the end rivets first slip and later yield before the working load is reached. In laboratory[30] tests the end rivets of large joints may break off entirely, after large plastic deformation, before the maximum joint load is reached. The ratio between the maximum load and the design load is considered the significant over-all factor of safety of the joint, but this maximum load is not arrived at until certain rivets use up at least a considerable portion of their ability to take plastic deformation.

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[29] Assistant Director, Fritz Engineering Laboratory, Lehigh University, Bethlehem, Pa.

[30] R. E. Davis, G. B. Woodruff, and H. E. Davis, "Tension Tests of Large Riveted Joints," *Transactions*, Am. Soc. Civil Engrs., Vol. 105, p. 1193 (1940).

The author has indicated the usefulness of ductility as accident insurance. The steel skeleton of a building during a hurricane or earthquake, and the end post of a highway or railway bridge during a wreck are examples where such ductility is put to good use and where danger to human life is involved.

The writer does not believe that cases needing high deformability are "decidedly rare in engineering practice" but agrees thoroughly with the need for "more direct and searching tests" of structures and structural elements. The writer believes that there is, in general, a "case" for present ductility requirements. Mr. Gillett has pointed out, however, the unjustness of specifying slight variations in ductility requirements for different metals going into similar services. Mr. MacGregor, in another paper of this symposium, has differentiated between ductility measured before and after necking down commences. This may be a very significant differentiation with respect to what is actually needed in a structure.

Mr. Gillett has focused attention on an important subject which needs further study. The significance of the ductility features may in some applications be limited, but in other cases the writer believes the question remains an open one.

MR. L. J. LARSON[31].—It is very gratifying to have someone of Mr. Gillett's standing in the field raise the question: "Why do we need so much ductility?"

For a number of years I have been associated with welding and have found that ductility is the subject of more discussions and arguments than any other property of weld metal. The minimum elongation specified for deposited metal has been increased constantly and at a rapid rate. The first code for welded pressure vessels specified that the weld metal must have a minimum elongation of 20 per cent in 2 in. and a strength equal to the minimum specified for the stock (about 60,000 psi.). The yield point was not specified but was approximately 45,000 psi. A little later, when high-strength steels came into use, the same elongation was required for welds having strengths over 70,000 psi. and yield points of about 50,000 psi. Now we have specifications which require a yield point of 50,000 psi. and elongations of over 30 per cent. The higher the requirements for any material, the more difficult and expensive it becomes to produce. The question arises: "Are such high requirements justified?"

Although weld metal is not produced in large tonnages, it is a vital part of many structures and is, therefore, an important engineering material, in which this Society should be interested. Engineers who use welding and prepare specifications for welded structures will rely to a large extent on the recommendations made by A.S.T.M.

Mr. Seely points out that some steels have yield ratios as high as 80 per cent. For weld metal such a ratio is quite common and a yield ratio of nearly 90 per cent is not unusual. This high ratio is the result of requiring such high yield points for weld metal. Whether the high yield point of weld metal is of any advantage is questionable, so long as the yield point of the weld is equal to that of the plate welded.

MR. L. H. FRY.[32]—It seems to me that in considering the acceptance tests and specifications, we should bear in mind the history of the acceptance test as indicated by Mr. Gillett. The practical engineer finds that a certain material gives satisfactory results in a service. He then makes a tension test, records the tensile properties and writes a specification boosting the tensile properties as far as his conscience and the manufacturers will let him.

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[31] Consulting Welding Engineer, Milwaukee, Wis.

[32] Railway Engineer, Edgewater Steel Co., Pittsburgh, Pa.

I am a little disturbed by a minor flaw that I find in both the papers by MacGregor and Seely. This Society gives definite definitions of yield strength and yield point, recognizing them as distinctive properties. Mr. MacGregor showed a definite yield point and called it yield strength. Mr. Seely showed yield strength and called it yield point. It seems to me that, of all people, the members of the American Society for Testing Materials should use definite terms to differentiate between different characteristics of the various materials.

MR. H. F. MOORE.[33]—Although at present the problem of strength and ductility under triaxial tension is well outside the range of "practical" study, it is one of great importance. Under balanced triaxial stress shearing stress becomes zero, and it seems that some sort of cohesive strength must be the dominating factor in resistance. Unfortunately, so far no triaxial tension test has been devised in which stresses can be accurately computed, and in which primary failure is due to triaxial tensile stress. The development of such a test would be a major technical achievement.

MR. R. L. TEMPLIN.[34]—I think we have all been impressed with the empirical nature of the tension test as brought out by these three papers. Furthermore, I do not see how we can get away from the rather empirical character of all of our mechanical property tests. The more refinements we put into them or think we should put into them, will of necessity depend on what we want to do with the values we get from the tests. I am particularly concerned about one of the points in Mr. Seely's paper, wherein he mentions the tendency of designers to decrease the magnitude of the offset in order to evaluate the yield strength of the material. In general, the material supplier and those who have to test the material on a routine mass production basis have to use the testing apparatus now available and therefore prefer to have a larger value for the offset so that they can more readily obtain results which will check with those obtained by the party who is buying the material and is going to retest it. In view of what we have seen brought out in these papers and in line with one of the time-honored customs of selecting design stresses in engineering structures, that is, dividing the tensile strength or yield point or yield strength by some factor in order to arrive at a design stress; it would seem perfectly logical that if we must have a value that corresponds to that nonexistent property, elastic limit, in our usual formulas, we could with impunity get at a value that would serve in lieu of this proportional limit or elastic limit by merely taking some percentage of the yield strength determined with a fairly large offset value, and be on just as safe ground as we have been in the past with the design of our engineering structure. I think it will be found that this procedure will give just as satisfactory structures as we have had in the past and in some instances such procedure may enhance their value. I am quite in sympathy with Mr. Gillett's remarks on ductility, yet I cannot find just how we are to use that value in engineering design. We insist that we have to have some ductility in certain structures and in others we think we ought to have a minimum ductility value as determined from the tension test, whether we really know how to use the value or not. In defense of the tension test and the values we obtain from it, I think it is only fair to state that we still believe that the test and the values it gives serve a very useful purpose in the control of metal products and the fabrication procedures that are used in making these products. They serve as a satisfactory yardstick, so to speak, to make changes in fabrication processes, to develop new alloys or new processes, and they serve as one of the items in the specifications governing products to help insure that the immediate lot of material corresponds, at least approximately, to previous lots furnished or produced. Our tendency is to narrow down or further qualify the limitations imposed on obtaining these values. We have had a paper on the effects of speed of testing.[35] There is a tendency to define limits for speed of testing so that we can get closer checks on our values. We may some day come to the limiting of the temperatures involved in making the tension test. Certainly we know that temperature has an effect, but with all of these limitations, are we not expecting a lot from the results as given by the empirical tension test? When we come to use these values as one of the cornerstones in the design of engineering structures, it would seem to me that rather than try to expect so much from tension tests, the designer should know more about the material and the behavior of it in the structure, bringing into the picture the factors, other than the tensile properties of the material, that affect the service life and the utility of the structure. Two well-known and well-recognized factors in that category, of course, are the effects of structure fabrication processes and the design of the structure.

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[33] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[34] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

MR. C. W. MACGREGOR[36] (*Author's closure by letter*).—Mr. Osgood refers to the difference in trueness of the stress and strain definitions used in the paper. While the author is in agreement generally with Mr. Osgood's comments, he would like to point out again that basically a certain ambiguity in the choice of the proper reference length exists in the definition of strain given by $\epsilon_o = \frac{L - L_o}{L_o}$ which does not exist where the strain is defined as $\epsilon = \log \frac{L}{L_o}$. In the latter case, the change in length is always referred to the length from which that change is produced and not to the original length. In this respect then the definition of ϵ fits in perhaps better with our essential ideas of strain. The most important advantages in its favor lie in the ease with which ϵ may be obtained from area measurements in a necked-down test specimen, in the convenient expressions for ductility, and in the resultant shape of the stress-strain curve so constructed.

The method of plotting the true stress as a function of q_o is discussed by Mr. Peterson. As had been found by others earlier, and also verified by our own experiments, the portion of this curve constructed from the maximum load point to fracture curves gradually upward, and is not a straight line in this region. Attempts have been made in the past to extrapolate the tangent of this curve at the maximum load point and to extend it to a value of $q_o = 1$. The value of stress derived in this manner represents a fictitious stress which would not be received in the material even if the reduction of $q_o = 1$ were realized. For this reason, the physical significance of this stress value is not apparent. It is also possible to show analytically that if such a straight line were used, the true stress at the maximum load is half that at $q_o = 1$. Tests have shown that such a relation is realized only with a ductile material in the annealed condition.

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[35] Paul G. Jones and H. F. Moore, "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals," see p. 610.

[36] Associate Professor of Applied Mechanics, Massachusetts Institute of Technology, Cambridge, Mass.

In the work of Stead,[37] the true stress is plotted as a function of decreasing diameter. Following this method, the true stress is related linearly to the decreasing diameter in the region from the maximum load point to fracture. Experiments conducted by the author some time ago[38] showed that straight lines were obtained with the same data in the necking region by both the Stead method and also by the $S - q'$ procedure. It was shown for the range of values of $\frac{d}{d_o}$ received in the tension test, that a plot of $\frac{d}{d_o}$ *versus* $2 \log \frac{d}{d_o}$ is a straight line. It is then apparent why both methods yield a straight line in this portion of the curve.

It is possible now to show analytically that if the straight line in the Stead method is extrapolated to zero diameter, the true stress corresponding to the maximum load is one-third of the true stress at zero diameter instead of one-half as predicted by the $S - q_o$ method. This procedure would appear, at first glance, to be better than that used by Ludwik and others since the true stress-diameter curve from the maximum load point to fracture follows the tangent at the maximum load, whereas, in the $S - q_o$ method it does not. The value of stress at zero diameter has been called by O'Neill and Greenwood[39] the "pressure of fluidity." The assumption of a straight line for small diameter values in this case is, however, also open to question. Test results obtained by the author some time ago show values of true stress corresponding to zero diameters for annealed ductile metals varying from 2.75 to 2.82 times those at the maximum load. Work-hardened or heat-treated steels do not check this relation as was found in the $S - q_o$ procedure. The Stead method also is not readily applicable to flat bars and the abscissa is not a strain.

The author thoroughly agrees with Mr. Peterson's comments on the possibility of obtaining more significant results in the correlation of fatigue data by using true stress and strain values. It is hoped that Mr. Peterson will publish the results of such a correlation in the near future.

In answer to Mr. Lester's question as to whether there is a definite relationship between the true breaking stress and the cohesive strength of the material, the breaking strength is approximately "the technical cohesive strength" of the cold-worked metal.

Concerning the distinction between yield strength and yield point mentioned by Mr. Fry, reference is hereby made to page 509 of the paper.

In closing, the author would like to thank all those who have contributed to the discussion. It is hoped that more research work will be done in future in the various laboratories in the country on true stress-strain relations in the tension test. It is felt that the tension

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[37] J. Stead, "The Cold-Working of Steel with Reference to the Tensile Test," *Journal*, Iron and Steel Inst., Vol. 107, pp. 377-416 (1923).

[38] C. W. MacGregor, "Relations Between Stress and Reduction in Area for Tensile Tests of Metals," *Technical Publication 805, Metals Technology*, April, 1937; also, "Differential Area Relations in the Plastic State for Uniaxial Stress," Stephen Timoshenko Sixtieth Anniversary Volume, The Macmillan Co., New York, N. Y., (1939), and "A Two-Load Method of Determining the Average True Stress-Strain Curve in Tension," *Journal of Applied Mechanics*, Vol. 6, No. 4, December, 1939, pp. A156-158.

[39] H. O'Neill and H. Greenwood, "Observations on the Pressure of Fluidity of Annealed Metals," *Journal*, Inst. Metals, Vol. XLVIII, No. 1, pp. 47-68 (1932).

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test can still be developed much further than heretofore, and that a fruitful direction of research lies in the application of true stress-strain methods.

MR. F. B. SEELY[40] (*Author's closure*). —Before commenting on some of the questions raised in the discussions, it may be well to focus attention on the fact that the aim of the symposium was to emphasize the significance of the tension test *in relation to the design* of a member whose primary function is to resist forces (loads) applied to it. The significance of the test for this purpose may be only remotely related to the significance of the test for determining whether material should be accepted or rejected in accordance with specifications. In order to bring out how test results are used in a rational procedure of design, and thereby to present the basis on which the significance of a test is to be judged from the viewpoint of this symposium, the steps in a rational design of a member are outlined as follows:

Step 1.—The mode of probable failure of the member as used in the load-resisting structure, machine, or apparatus is determined. By *failure* is meant any action in the member, resulting from the applied loads, that would cause the member to cease to function satisfactorily in the structure, etc. Failure may consist of (*a*) elastic deflection, (*b*) permanent deformation of a considerable portion of the member (general yielding in contrast to localized yielding) of a small amount in some cases and of a relatively large amount in others; furthermore, the yielding may be a continuing deformation such as creep at elevated temperature, or a yielding at ordinary temperature that is accompanied by strain hardening which prevents continuing deformation at the same load, (*c*) fracture consisting of either a rather sudden breaking of the member or a progressive (fatigue) fracture starting as a minute crack, etc. This step presents difficulties in the design of some load-resisting members, but a design becomes largely empirical if knowledge for this step is lacking.

Step 2.—A relation (equation or formula) is found between the loads and the quantity (deflection; stress, either shearing or tensile and either the statistical or localized value; shear strain-energy; etc.) which is judged to be significant in the failure of the member—the quantity that would limit the maximum value of the loads if the loads were increased until failure occurred. In our present state of knowledge this step usually involves simplifying assumptions and approximations in obtaining design formulas even when the significant quantity to be expressed in the formula is known, which is not always the case.

Step 3.—By appropriate tests the maximum value of the significant quantity in the failure, as expressed in the formula of step 2, is determined. Certain general properties of the material are also found which are an aid to the judgment needed in step 4.

Step 4.—A working value of the significant quantity to use in the equation obtained in step 2 is selected as some proportion of the value found in step 3. But the working value is largely the result of experience, practice, commercial and legal considerations, and other conditions that cannot be expressed in mathematical form, although one's judgment in making use of experience is guided by a knowledge of the ductility and other properties of the material.

The steps listed above are, of course, over-simplified, but they will serve perhaps in emphasizing how the significance of a test of a material is primarily related to the problem of design of a load-resisting member. Now, the main question raised in the second paper in this symposium was: Under what conditions of use or under what mode of failure of a load-resisting member is the simple tension test (as specified in the standards of this Society) an appropriate test for determining the maximum value of the strength quantity that is associated with the failure, and hence that limits the loads that can be applied to the member?

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[40] Professor and Head, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

It was pointed out in the paper on the Strength Features of the Tension Test that the significance of the simple tension test for so-called ductile metals was limited largely to uses in which members fail by general yielding accompanied by strain hardening, although the amount of yielding constituting failure may vary from a small value to a relatively large one. Such a failure occurs when a member is made of ductile metal, is loaded at ordinary temperatures by static loads, and is subjected to a state of stress in which relatively large shearing stresses can be developed.

The paper by Mr. MacGregor and the discussion by Messrs. McAdam and Mebs are valuable contributions in showing possible methods of modifying the simple tension test (as usually made) and of interpreting the tension test data to reveal additional significant properties of materials. It is hoped that by such methods the tension test may be made significant in rational design procedure in a wider range of service conditions, perhaps under conditions in which localized actions are the significant actions associated with failure.

Mr. Osgood raises the question as to overlapping of conditions 3 and 4 under which tensile strength values are significant, p. 540. It is true that there is some overlapping, but it was felt that there was an essential difference between the two conditions, in that condition 3 does not involve localized action, whereas condition 4 is concerned mainly with the effect of localized action. A deep circumferential sharp V-notch in a cylindrical tension member would perhaps be considered to fall under condition 3, and a similar shallow notch would be considered to fall under condition 4.

Mr. Osgood also questions the statement "that in this discussion failure by elastic buckling is not considered," p. 540. His remarks are well considered in the light of his interpretation of the statement. But the idea meant to be conveyed by the author was entirely different from Mr. Osgood's interpretation. The sentence preceding the statement quoted above emphasizes the fact that strength values, such as yield point, etc., from the tension test are significant when the member fails by general yielding; these strength values, however, would not be significant if, prior to such yielding, the member failed because of excessive elastic deflection alone or because of such deflection that passes on into buckling action, and therefore attention was called to the fact that this type of failure was not included in the discussion.

The discussion by Mr. Jones will tend to raise doubt in the mind of those who like to consider yield point as being an inherent property of the material in contrast to yield strength which is classed as an arbitrary value since the offset used in determining it is arbitrarily specified. In this connection, attention should be called again to the fact that a yield point determined in a tension test specimen in which the stress is uniformly distributed may have doubtful meaning, in fulfilling the requirement in step 3 of the above design procedure, if the material is used in a member in which the state of stress is more complex; for example, a beam made of the material will show no yield point. The question then is raised as to whether, in step 3 of the above design procedure, a tension test of a ductile metal is an appropriate test for so simple a member as a beam.

Mr. Smith gives convincing evidence that a change in the mode of failure of heat-treated steel from a (ductile) yielding type of failure to a brittle fracture type may take place with a change in the ratio of the steady stress to the alternating stress as a member is subjected to repeated stress of varying ranges of stress. His discussion emphasizes the importance of the knowledge of the mode of failure as a first step in the procedure for a rational design, as indicated at the beginning of this closing discussion.

Mr. Riegel also presents valuable evidence of the importance of a study of failures as a means of determining appropriate tests for indicating the suitability of materials for service.

Mr. Johnston's illustration from Neuber's work is very welcome. More work of this nature needs to be done on the mechanics of notches. Likewise his observation that the work of Kuntze on cohesive strength needs further study and corroborating is very appropriate. It is an encouraging sign also to observe in Mr. Johnston's discussion that attempts are being made at various places to devise tests that are really significant under various conditions of service.

The interesting test described by Mr. Collins also is an attempt to devise a test which may bring out properties not revealed by usual tests.

Mr. Rutledge brings out well the practical problem of making tests serve a dual function; he also suggests indirectly the increasing demand of design engineers for properties corresponding to more refined measurements. This idea is referred to in Mr. Templin's discussion and is treated later in this discussion.

Mr. Fry's comment that a yield strength was shown but was called a yield point refers to a diagram used as a slide in the oral presentation of the paper but not published as part of the paper. In the paper as published the terms are used as defined by this Society.

Mr. Moore points out the importance of the effect of triaxial tension on strength and ductility properties and adds that "no triaxial tension test has been devised in which stresses can be accurately computed. . . .," and that "The development of such a test would be a major technical achievement." In fact, there would be considerable value and satisfaction in merely knowing whether such stresses exist under certain conditions. For example, do they frequently exist at grain boundaries of a stressed member particularly at the junction of three or more crystals? Do they exist at certain types of abrupt changes of section (see Mr. Johnston's discussion) or at the edges of internal flaws? Are they partly responsible for fatigue fractures? Even though they cannot be calculated it would be a great help to know under what conditions they are likely to change the mode of failure of a member from ductile yielding to brittle fracture. Perhaps further analyses and tests similar to those by Neuber and Kuntze may yield fruitful results.

Mr. Templin calls attention to the tendency of designers to require values of yield strength corresponding to very small offsets. To determine the significance of very small offsets we must go back to step 1 in the procedure for design as given at the beginning of this closure. What action in the member causes it to fail—to cease to function satisfactorily? If yield strength based on a relatively large offset (0.1 to 0.2 per cent) is considered to be the maximum utilizable strength of the material, the member must be assumed to fail by general yielding because a large offset is a fairly accurate measure of the actual permanent set or "yield." If, on the other hand, a yield strength corresponding to a very small offset (0.001 to 0.01 per cent) is considered to be the upper limit of useful strength of the material, it is not clear what is considered to be the action in the member that causes it to cease to function satisfactorily and hence to fail. It cannot be assumed in general that such small offsets are equal to the permanent set or "yield"; neither can it be assumed, without evidence furnished by special tests in addition to the usual tension test, that they represent deviations from the stress-strain curve within the elastic action of the material. In fact, in this region of small offsets we are dealing with an uncertain and indefinite no-man's-land condition which is difficult to relate, by use of the simple tension test alone, to a definite mode of failure. The use of a small offset in the control of metal processing, as for example in determining whether a heat treatment has been carried out satisfactorily in stabilizing the material, is not here under discussion.

In closing, the author wishes to express his appreciation to all those who have contributed so liberally to the discussion. An attempt to get at the real meaning and significance of test results promises much for the future.

MR. H. W. GILLETT[41] (*Author's closure*).—Mr. Seely's closure constitutes the best answer to Mr. Tuckerman. In some earlier correspondence Tuckerman mentioned that a perfectly analogous case was Grade A and Grade B milk; that irrespective of whether he could stand a lot of "bugs," by gum he was going to buy Grade A milk according to A.S.T.M. specifications to hold the bug content down. In reply, I pointed out that one member of my family has been instructed by the family doctor not to drink Grade A or Grade B, but, instead, to drink buttermilk. I do not know anything about bacteriology, but I am quite sure there are more bugs, of one kind or another, in buttermilk than in Grade A milk, so that for some circumstances your ordinary test may give you a sign pointing in the wrong direction. Mr. Tuckerman also brought up his duralumin with only 12 per cent elongation; where he was in a position to ride over the old specifications, he was willing to reduce the specification to 12 per cent because he had studied the material and thought it was good stuff. He tells you that 7-yr. experience thoroughly justifies his judgment. There may well be many other similar cases, but the poor son-of-a-gun who has to buy a material on specifications that are sacred and taken as such, has no chance to exercise such intelligence. I still say that when we do not know why we draw a certain provision in a specification as we do, we ought to put in a footnote saying that we *do not* know.

MR. LESSELLS (*Chairman's closure*).—It was stated in the Foreword that the tension test is one of the oldest and most important tests. Although this is true the papers and discussions contained in this Symposium indicate that research is extending our frontier of knowledge even to this well-known form of test and suggest much information yet remains to be uncovered.

The purpose in holding it was "to allow clarification of certain of the tensile characteristics" as an aid to the designer and it is to be hoped that the material presented has done this, at least in part.

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AN INVESTIGATION OF THE EFFECT OF RATE OF STRAIN ON THE RESULTS OF TENSION TESTS OF METALS

By Paul G. Jones[1] and H. F. Moore[1]

Synopsis

Results of tension tests of 14 metals are reported using different speeds of testing. The apparatus used was autographic and employed a duplicate apparatus for weighing load—a compression block with carbon-pile compressometer and, in series with it, a steel tension weigh bar with an electric resistance extensometer. Stretch of specimen was measured by means of an electric resistance extensometer. Time records were made photographically on an oscillograph film by contacts fitted to an electric clock.

The results of the tests showed appreciable effect of rate of strain on the tension test results, but, except for the stainless steel specimens, the amount of this variation might be expressed by the statement that a variation of rate of strain from N per cent per minute to $2N$ would introduce a variation not greater than 1 per cent in value of yield strength or tensile strength and not greater than 3 per cent in elongation or reduction of area, with the exception of elongation and reduction of area for the brittle cast metals tested.

The second part of the paper gives proposed methods for determining rates of strain in tension tests using a type of testing machine in common use, not equipped with any special control apparatus. An appendix contains a discussion of the probable relative effect of variation between results from duplicate tests and of variation due to effect of speed on the ratio of rate of stretch of specimen to rate of speed of head of testing machine.

Problems of the proper speed of application of load to specimens have caused testing engineers concern for many years. There is a general notion that increased speed of straining increases strength values and diminishes ductility values, at least for metals. For very soft metals, such as lead, rate of straining is a very important factor in determining mechanical properties, and it seems to be of importance in testing cast iron, timber, concrete, and probably textiles. It may be one of the important factors in studying the strength and toughness of plastics.

At the 1936 meeting of this Society, Jones and Richart presented a paper on "The Effect of Testing Speed on Strength and Elastic Properties of Concrete."[2] In 1939 Committee E-1, Section on Effect of Speed of Testing, began an investigation of effect of speed of testing on the tension test results for metallic materials, following the general method used by Jones and Richart, and carried on by Jones in the Talbot

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II p. 380 (1936).

Materials Testing Laboratory of the University of Illinois under the immediate supervision of Prof. H. F. Moore.

Acknowledgment:

Funds for the investigation were provided in part from the A.S.T.M. Research Fund and in part by various industrial firms. The following furnished money, material, or both:

Dow Chemical Co.,
National Smelting Co.,
Aluminum Company of America,
Baldwin-Southwark Corp.,
American Brass Co.,
Westinghouse Electric and Manufacturing Co.,
General Cable Corp.
Bridgeport Brass Co.,
Chase Brass and Copper Co.,
International Nickel Co.,
Jones & Laughlin Steel Corp., and
University of Illinois.

The investigation was organized as a cooperative investigation sponsored by the A.S.T.M. and the University of Illinois. It is a part of the research program of the Illinois Engineering Experiment Station, M. L. Enger, Director, and is carried on in the Department of Theoretical and Applied Mechanics, of which F. B. Seely is head.

Speed Effect in Material and in Testing Apparatus:

The effect of speed of loading or of rate of strain on the results of ordinary tension tests may be separated into three parts: (1) variation in results due to inertia effects in the testing machine or in the strain-measuring apparatus, (2) variation due to personal equation of the testing machine operator, and (3) variation in the tensile properties of a material under different rates of strain. It is the third of these causes of variation which is to be studied. Very little can be said about the personal equation of testing machine operators, except that it may cause a considerable deviation of results, especially in testing machines with a beam-balance weighing mechanism, which is the type of testing machine still most widely used in the United States, although machines with self-indicating weighing devices are fast coming into use. To avoid variations due to personal equation of operator the testing machine used in a study of effect of rate of strain should be fitted with a self-indicating and recording weighing mechanism.

There is a good deal of experience available to show that the rate of strain in a specimen for a given nominal speed of head of testing machine varies appreciably for different testing machines, for different types of grips, for different grips of the same type, for different materials, and for different types and lengths of specimens.

Since, then, the most important object of the tests described in this paper was to investigate the effect of *rate of strain* upon the yield strength, tensile strength, elongation, and reduction of area of tension test specimens of a variety of metals, the testing apparatus used gave autographic records on a time base for loads and stretches and was designed to reduce inertia of parts of the measuring and recording apparatus to a minimum.

Materials and Specimens:

A.S.T.M. standard $\frac{1}{2}$-in. tension specimens[3] with a 2-in. gage length were used in all the tests to determine effect of rate of strain. Tests were made on specimens of the metals shown in Table I.

Description of Tests:

The tests were made in a 200,000-lb. capacity Riehlé screw-power testing

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[3] Standard Methods of Tension Testing of Metallic Materials (E 8 – 36), 1939 Book of A.S.T.M. Standards, Part I, p. 750.

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machine, using the available free running head speeds of 0.05, 0.40, 1.0 and 8.0 in. per min.

The elongation of the specimen over a 2-in. gage length was measured by a strain gage shown in Fig. 1, which measures the change in drop of potential which occurs when a contact is moved along a resistance wire. Resistance wires were mounted to each of two insulating blocks, which were in turn fastened to opposite sides of a yoke by means of steel springs. The yoke was fastened to the specimen with three pointed screws, one of which was pressed against the specimen by a coil spring so that as the cross-section of the specimen diminished under axial tensile load the extensometer would not fall off from the specimen. The resistance wires pressed against contacts which were fastened to, but insulated from, another yoke which was also fastened to the specimen with three pointed screws. Thus as the specimen elongated, increasing the distance between the yokes, the contacts were allowed to slide over the resistance wires which, when connected to a voltage supply, would produce a changing potential at the contacts. This potential was measured by means of an oscillograph. It was found that the resistance wires used were very uniform in resistance per unit of length, resulting in a linear relationship between change in potential at the contacts and displacement of the contacts. The sensitivity of this gage was very flexible, since it was a function of the voltage supply used and of the sensitivity of the oscillograph element. With the highest sensitivity used in obtaining yield strength of some of the non-ferrous metals, a strain of 0.00005 in. per inch (0.005 per cent) was represented by 0.01-in. ordinate on the oscillograph record. Successive calibrations at this sensitivity showed less than

TABLE I.—METALS TESTED IN INVESTIGATION OF EFFECT OF SPEED OF TESTING.

Metal	Approximate Chemical Composition, per cent												
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Chromium	Magnesium	Iron	Aluminum	Nickel	Zinc	Lead
Low-alloy steel (Corten)	0.08	0.27	0.145	0.020	0.80	0.41	1.01	....	remainder	....	....	....	....
S.A.E. 1045 steel	0.44	0.75	0.014	0.021	0.21	....	....	....	remainder	....	....	....	....
S.A.E. 1020 steel	0.20	0.45	0.045	0.055	....	....	....	....	remainder	....	....	....	....
Stainless steel (18-8)[a]	0.20	0.70	0.125	....	0.35	....	18.0	....	remainder	....	8.0	....	....
Electrolytic copper, commercially hard	....	....	....	....	....	99.95+	....	....	....	....	....	....	....
Copper, hot-rolled	....	....	....	....	....	99.95+	....	....	....	....	....	....	....
Monel metal, annealed, cold-drawn	0.15	1.00	....	....	0.10	30.00	....	....	1.40	....	67.0	....	....
Duralumin, 17S-T	....	0.61	....	....	0.49	4.19	....	0.50	0.25	remainder	....	....	....
Sand-cast aluminum alloy Chill-cast aluminum alloy	....	0.3	....	....	5.0	0.4	....	0.05	0.8	remainder	....	....	[b]
Drawn magnesium alloy (Dowmetal J)	....	0.28	....	....	....	....	....	remainder	....	6.50	....	1.12	....
Cast magnesium alloy (Dowmetal H)	....	0.22	....	....	....	....	....	remainder	....	6.02	....	2.72	....
Brass, soft temper	....	....	....	....	....	69.69	....	....	0.01	....	....	30.23	0.07
Brass, drill temper	....	....	....	....	....	69.71	....	....	0.01	....	....	30.21	0.07

[a] Probably contains a small content of selenium or molybdenum.

[b] Titanium, 0.2 per cent.

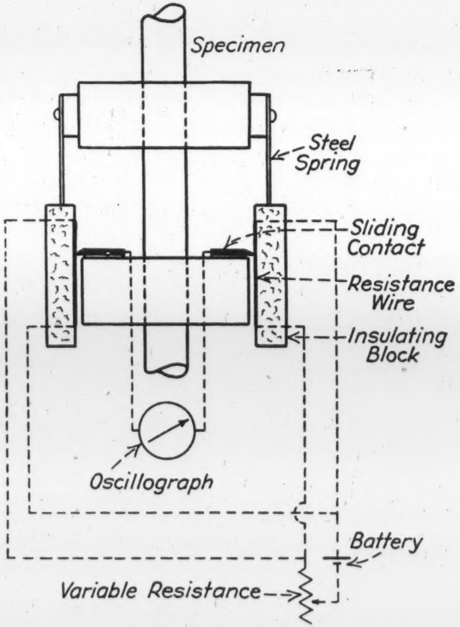
Specimen
Steel Spring
Sliding Contact
Resistance Wire
Insulating Block
Oscillograph
Battery
Variable Resistance

FIG. 1.—Strain-Measuring Apparatus.

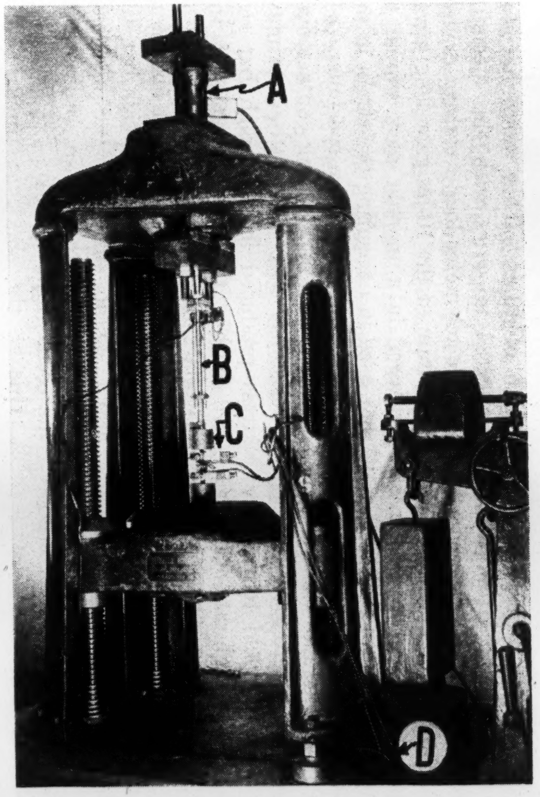

FIG. 2.—200,000-lb. Riehlé Testing Machine Rigged for Measurement of Rate of Loading and Rate of Strain.

A. Compression block for measuring load, fitted with a Peters carbon-pile telemeter for measuring lateral expansion under load.
B. Tension weighbar fitted with a resistance-type extensometer.
C. Resistance-type extensometer on specimen.
D. Wires leading to batteries and oscillograph.

1 per cent variation. The strain gage remained on the specimen through rupture, and frequent calibrations were made to insure proper adjustment.

The rate of change in strain-rate in the 2-in. gage length even for the highest speed of head used is not very great and it is felt that there is little danger of inertia effects entering into the measurement of strain. However for load measurement it is different. The transition from the elastic (zero load to yield) to the plastic state (yield to fracture) of mild steel causes a high rate of change in load rate. (There is probably also a high rate of change in *unit* strain rate in localized parts of the specimen but not over the 2-in. gage length.) This high rate of change in load rate at the yield point of mild steel may cause considerable error in measurements of load taken by the apparatus in which there is appreciable inertia effect.

For this reason two independent methods of measuring load were used, each of which, it is felt, involves very little inertia effect. In one method a telemeter pressure block was employed. The telemeter gage consists of a series of contacts between a pile of thin carbon disks which when compressed undergo a measurable change in electrical resistance. This change in resistance is utilized in a simple Wheatstone bridge circuit. The displacement of the disks is produced by the lateral expansion of a steel cylinder which transmits the load. Since the loading block operates in compression, it was necessary to use a saddle arrangement to obtain tension in the specimen (Fig. 2). The sensitivity of this block could be changed by means of shunts across the oscillograph element. The maximum sensitivity used with this block was about 30-lb. load on the specimen (corresponding to 0.01-in. ordinate on an oscillograph record).

The second method of measuring load consisted of a steel weigh bar 18 in. long with a reduced section over a gage length of 10 in. This weigh bar was connected in series with the specimen and the telemeter load-weighing device. The strain in the 10-in. gage length was measured in a manner very similar to that used in measuring the strain in the 2-in. gage length on the specimen, that is, by the use of a sliding contact over a resistance wire. Since the maximum stress produced in the weigh bar was well within the elastic range of the material, load was proportional to the stretch of the weigh bar. The maximum sensitivity used with the weigh bar was about 25 lb. load on the specimen (corresponding to 0.01 in. ordinate on the oscillograph record).

Due to the shock at fracture of a specimen, frequent calibrations of the two load-measuring devices—telemeter and weigh bar—were deemed necessary. Calibrations were made against the load indicated by the weighing beam of the testing machine, which was found to be sensitive to 10 lb. In addition to these over-all calibrations, several check readings were taken on both telemeter and weigh bar by loading a dummy specimen before each test of a regular specimen.

The difference in strength values as obtained from the two load-measuring devices averaged about 2 per cent (1 per cent from the mean value). There was found to be no definite increase in the difference between the strength values indicated by the two gages with an increase in loading rate; therefore, if the readings of the load gages are affected by speed of testing then this effect must be approximately the same for each gage. However, it is hardly likely that two gages utilizing different principles of operation would be subject to the *same* speed effect. It is concluded that neither gage is appreciably affected by the rate at which it is loaded.

The load and strain measurements were recorded by means of a four-element oscillograph equipped with super-sensitive galvanometer elements of the oil-well type. The damped frequency of these elements is about 250 cycles per second. For the wave fronts produced in these tests the amount of over-shooting was negligible. It is realized, of course, that this method of recording would be unsuitable for extremely high rates of loading. The camera recording the deflection of these elements was driven by a 12-v. d.c. motor to provide the speeds of 0.35 to 200 in. per min. used in the tests. The light source used consisted of a 6 to 8 v., 2.5 amp. gas-filled oscillograph lamp which gave sufficient intensity of light to register on grade A Insurance Bromide paper. A timing wave on the oscillograph record was produced by an electric clock fitted with contacts giving impulses at intervals of 0.02 min. For the higher speeds, a 60-cycle alternating current was used as a timing wave.

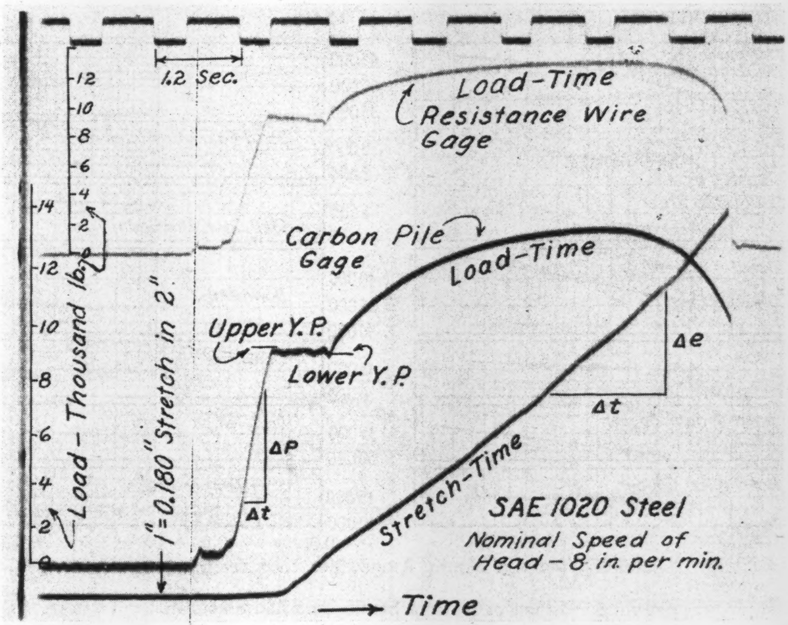

Fig. 3.—Typical Oscillograph Record.

Oscillograph Records of Tests:

Figure 3 shows a typical oscillograph record for a S.A.E. 1020 steel tested at a nominal head speed of 8 in. per min. The timing wave is at the top of the diagram, each impulse representing 1.2 sec. as noted. The curves represent load-time and stretch-time relations. If the stretch-time relation were linear, the two load-time diagrams would also represent load-deformation. However, the stretch-time diagram is not a straight line as can be noted but approximates a straight line in the plastic region.

For those steels which had well-defined yield points a sensitive strain gage was not needed. In obtaining yield strengths of the non-ferrous metals, some tests were run in which a more sensitive strain gage and higher camera speeds were used. For these tests over-all load-time and stretch-time curves were not obtained. In other tests the strain gage was modified so that its sensitivity was automatically changed after the yield strength had been passed. This change in sensitivity of the strain gage was achieved by making electrical connection to the resistance wire at the position of the sliding contact when the yield strength was passed. By arranging the electrical circuit so that part of the wire carried considerably higher current than the other part, the two sensitivities were obtained.

By taking simultaneous readings of load deflection and strain deflection from the oscillograph records and referring to the proper calibration curve, data for stress-strain diagrams were obtained.

Relation of Properties to Rate of Strain: Yield Point and Yield Strength.—

Figure 4 shows the relation between the yield point and yield strength of the various metals and the rates of strain in the elastic region. This rate of strain represents the average rate in the region from about one-half to three-fourths the yield strength value for the different materials. For convenience in plotting the wide ranges of strain rate, a logarithmic scale is used for abscissas. The general trend shows an increased yield strength with increase in strain rate. Exceptions to this are the soft-temper brass and the duralumin which show no definite change and the cast Dowmetal which indicates a *decrease* in strength with increased strain rate. Concerning the yield point of the steels there is a greater difference in upper and lower yield point for the Corten than for the other steels. The Corten was tested without the use of the weigh bar load gage. It is noted that there is less effect of speed on the lower yield point than on the upper yield point.

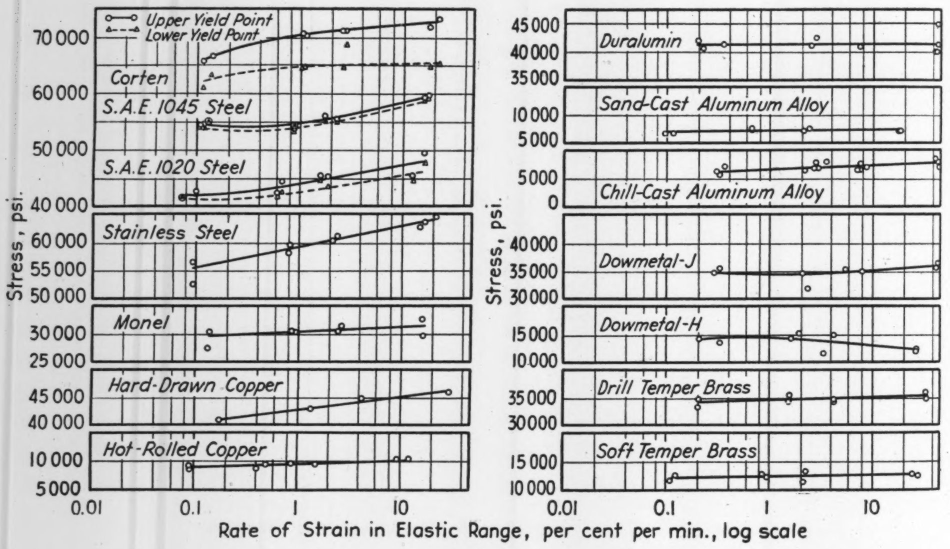

FIG. 4.—Effect of Rate of Strain on Yield Strength.

Tensile Strength.—Figure 5 shows the relation between the ultimate tensile strength and the rate of strain at the ultimate strength. The rate of strain was found from the slope of the strain-time diagram in the region of the ultimate strength. With the exception of the drawn and the cast Dowmetal, all of the materials show an increased strength with increase in strain rate. The relatively low strength of the cast Dowmetal under high speed may be explained by microscopic flaws in the material.

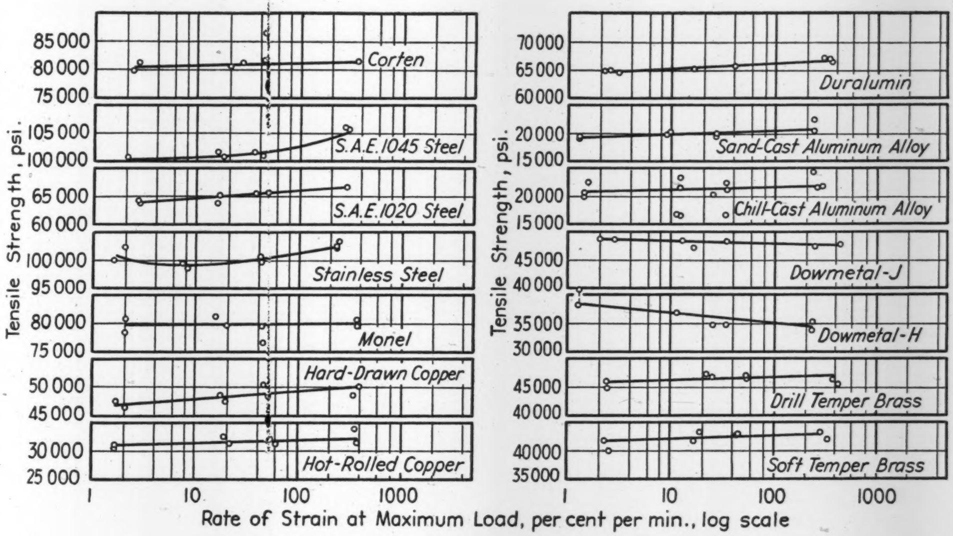

FIG. 5.—Effect of Rate of Strain on Tensile Strength.

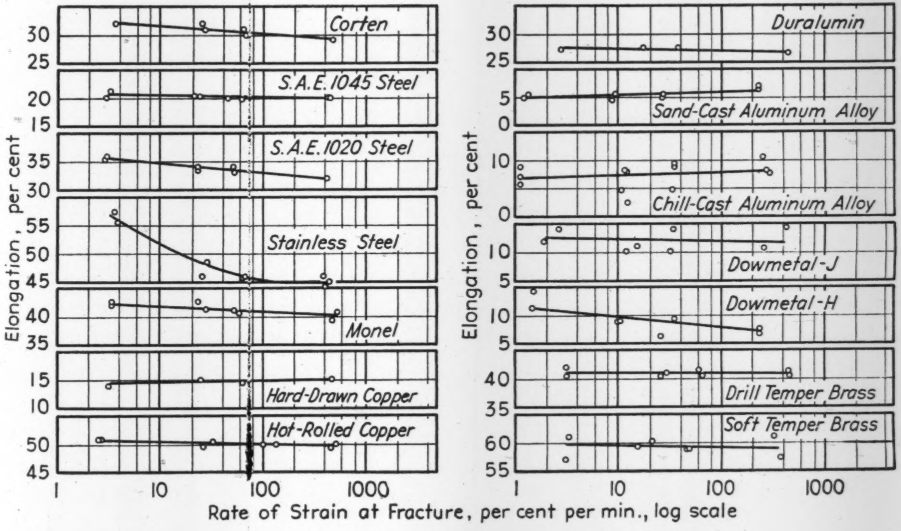

FIG. 6.—Effect of Rate of Strain on Elongation.

Elongation.—The relation between percentage elongation and rate of strain at fracture is shown in Fig. 6. The rate of strain was obtained from the slope of the strain-rate curve immediately before fracture. For the materials which showed no "necking down," this rate of strain was approximately the same as that at the ultimate strength. For the other materials it was higher, and for some the rate in inches per minute for the 2-in. gage length was even higher than the nominal speed of head. This, however, can be expected since the load

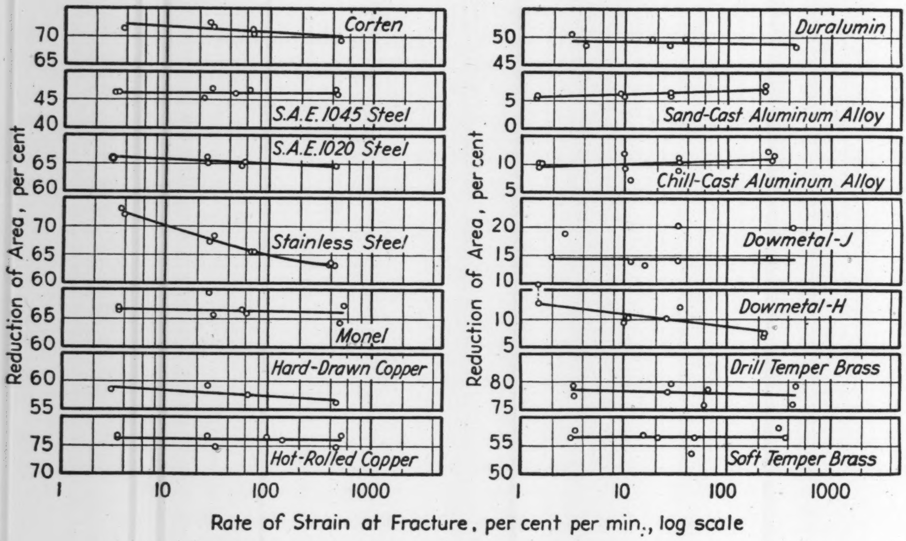

FIG. 7.—Effect of Rate of Strain on Reduction of Area.

TABLE II.—PERCENTAGE CHANGE IN MECHANICAL PROPERTIES FOR CHANGES OF TWO-FOLD (2×), FOUR-FOLD (4×), AND EIGHT-FOLD (8×) INCREASE IN RATE OF STRAIN.

Metal	Upper Yield Point, psi.			Yield Strength, psi.[a]			Tensile Strength, psi.			Elongation in 2 in., per cent			Reduction of Area, per cent		
	2×	4×	8×	2×	4×	8×	2×	4×	8×	2×	4×	8×	2×	4×	8×
Low-alloy steel (Corten)	0.9	1.7	2.6	0.5	0.9	1.4	0.2	0.4	0.5	1.5	2.9	4.4	0.4	0.8	1.3
S.A.E. 1045 steel	0.5	0.9	1.4	0.5	0.9	1.4	0.4	0.7	1.1	0.2	1.5	2.2	0	0	0
S.A.E. 1020 steel	1.3	2.6	3.9	0.9	1.8	2.8	0.6	1.1	1.8	1.6	3.2	4.8	0.3	0.7	1.0
Stainless steel (18-8)	....	....	....	1.8	3.6	5.4	0.5	0.9	1.4	3.7	7.4	11.0	2.3	4.1	6.8
Copper, hard-drawn	....	....	....	1.3	2.6	3.4	0.8	1.7	2.5	0.5	1.0	1.5	0.6	1.1	1.7
Copper, hot-rolled	....	....	....	1.7	3.4	3.9	0.6	1.1	2.5	0.3	0.5	1.5	0	0.1	1.7
Monel metal, annealed cold-drawn	....	....	....	0.8	1.6	2.4	0.1	0.2	0.2	0.7	1.4	2.2	0.1	0.2	0.4
Duralumin, 17S-T	....	....	....	0	0	0	0.5	0.9	1.4	0.5	0.9	1.4	0.2	0.3	0.5
Sand-cast aluminum alloy	1.1	2.1	3.2	....	....	....	1.0	2.1	3.2	2.4	4.9	7.4	2.6	5.2	7.8
Chill-cast aluminum alloy	....	....	....	2.4	4.7	7.1	0.7	1.4	2.2	2.4	4.7	7.1	2.3	4.5	6.8
Drawn magnesium alloy (Dowmetal J)	....	....	....	0.3	0.7	1.0	0.3	0.6	1.0	0.3	0.7	1.0	0	0	0
Cast magnesium alloy (Dowmetal H)	....	....	....	1.4	2.8	7.3	2.4	4.9	4.2	0.6	1.2	1.8	6.6	13.2	19.9
Brass, soft	....	....	....	0.2	0.5	0.7	0.5	0.9	1.4	0.5	1.0	1.4	0.1	0.3	0.8
Brass, drill temper	....	....	....	0.4	0.9	1.3	0.4	0.8	1.3	0.1	0.2	0.3	0.2	0.4	0.6

[a] For the low-alloy steel the S.A.E. 1045 steel and the S.A.E. 1020 steel the lower yield point and the yield strength are considered to be the same. Yield strength for the stainless steel and for the non-ferrous metals was determined by an offset of 0.2 per cent from the straight-line portion of the stress-strain diagram (except for hard-drawn copper for which an offset of 0.1 per cent was used).

was rapidly decreasing causing elastic recovery in the machine, weigh bar, etc. The elongation was found from a measurement of the gage length after rupture. The cast aluminum alloys and the hard-drawn copper show increased elongation while all the other materials show a decreased elongation with increase in strain rate.

Reduction of Area.—Figure 7 shows the relation between reduction of area and rate of strain at fracture. The reduction in area for any one material follows the same general trend as does the elongation.

Results of Tests at Four Different Speeds of Testing:

It is evident from a glance at Figs. 4 to 7, inclusive, that a large change in rate of strain causes a relatively small change in the tension test results for the metals tested. That means that in any specifications for rate of strain to be allowed for any test wide tolerance for deviations from the precise rate desired will be allowable. From the data shown in Figs. 4 to 7, inclusive, Table II has been compiled, showing the percentage change in the properties of the metals tested due to doubling, quadrupling, and increasing eight-fold the rate of strain. The values in Table II were computed on the assumption that for speeds within the range of reliable action of the weighing mechanism the change of properties was proportional to the logarithm of the rate of strain. The illustrations show that this is quite closely true up to head speeds of 2 in. or over, and for accurate behavior in tests of metals on a commercial testing machine speeds should not be higher than this, else the weighing beam cannot be kept in proper balance, or, for a self-indicating weighing mechanism the pointer is likely to move "jumpily."

Again examining Table II it is seen that for the steels tested the largest variations of values for varying rates of strain are found in the upper yield point —the yield point as determined by the "drop of the beam." For steels which have a yield point, the lower yield point and the yield strength as determined by an offset from the straight-line part of a stress-strain diagram may, without serious error, be regarded as having the same value. The elongation in 2 in. seems to be more affected by change of rate of strain than any other of the properties, while the reduction of area is slightly affected even by an eight-fold increase in rate of strain.

It is not the function of the authors to specify what tolerances are suitable for rates of strain. They would point out, however, that for the steels, if it were specified that the rate of strain should be not less than N per cent per minute and not greater than $2N$, the effect on test results would be small and that it would not be very serious even for a tolerance from N to $4N$. Tensile strength values and reduction of area values for the steels tested would not be very seriously affected even if the limits of rate of strain were set at N and $8N$ per cent per minute, respectively.

Examining the changes in properties of the non-ferrous metals tested, the values of yield strength seem relatively little affected by change of rate of strain, nor do the values for tensile strength and elongation in 2 in., with the exception of the cast aluminum alloys and the cast magnesium alloy, whose elongation seems to be relatively sensitive to changes in rate of strain. The reduction of area for these three metals seems to be seriously affected by change of rate of strain.

Relation of Speed of Head of Testing Machine to Rate of Strain:

The custom of judging speed of testing by speed of head of testing machine has become pretty well fixed in the minds of American testing machine operators. This is largely because at the present time the majority of testing machines in commercial service in this country are screw-power machines with a balance beam for weighing the load. The speed of head is fixed by the train of gears used, and usually there are four, six, or eight speeds of head available, through gear changes. The speed of head can be readily predicted for a screw-power machine, but not very closely controlled. By way of contrast in a hydraulic press type of testing machine the speed of head can be very closely controlled but not so accurately predicted as in a screw-power machine in good mechanical condition.

TABLE III.—RATIO (*R*) OF RATE OF STRETCH OF SPECIMEN IN GAGE LENGTH TO SPEED OF HEAD OF TESTING MACHINE.

Each tabulated value is the average of two test results on duplicate specimens.
Values of *R* are given in per cent.

Testing Machine	Nominal Speed of Head, in. per min.	Ratio of Rate of Stretch to Speed of Head									
		S.A.E. 1020 Steel[a]		S.A.E. 1045 Steel[a]		Duralumin (17S-T)[a]		Hard-Drawn Brass[a]		Hard-Drawn Copper[a]	
		Elastic	Plastic	Elastic	Plastic	Elastic	Plastic	Elastic	Plastic	Elastic	Plastic
SPECIMENS HELD IN WEDGE GRIPS, 8-in. Gage Length											
100 000-lb. Riehlé	0.10	6.21	55.0	8.47	44.7	13.40	36.1	10.80	10.0	10.50	25.0
	0.61	6.41	42.3	8.77	44.9	13.40	50.6	12.70	25.4	10.90	38.1
100 000-lb. Olsen	0.05	2.80	52.6	4.31	53.3	8.54	35.7	7.36	48.6	5.78	18.3
	1.04	3.17	53.0	4.38	54.1	8.26	50.4	7.44	45.3	5.63	34.3
50 000-lb. Riehlé	0.05	4.41	52.9	6.29	51.6	12.00	49.4	10.90	....	9.08	36.6
	0.78	4.59	52.7	6.42	51.5	12.00	54.0	11.30	....	7.74	33.4
SPECIMENS HELD IN WEDGE GRIPS, 2-in. Gage Length											
100 000-lb. Riehlé	0.10	1.58	22.3	2.32	16.7	4.16	19.7	3.42	49.7	2.88	8.9
	0.61	1.58	17.4	2.38	19.6	3.82	23.4	3.64	11.6	2.70	9.1
100 000-lb. Olsen	0.05	0.75	26.4	1.13	28.7	2.37	10.5	1.90	31.2	1.54	20.9
	1.04	0.75	25.0	1.12	26.2	2.04	19.7	1.88	19.8	1.40	16.5
50 000-lb. Riehlé	0.05	1.10	24.2	1.57	21.2	3.22	16.4	2.99	....	2.16	10.4
	0.78	1.29	21.3	1.58	22.0	2.90	16.9	0.99	....	1.66	4.4
SPECIMENS HELD IN SHOULDERED GRIPS, 8-in. Gage Length											
100 000-lb. Riehlé	0.10	12.0	96.6	18.0	97.6	30.0	80.8	29.1	100.0	24.0	89.1
	0.61	14.6	88.1	14.7	93.2	26.4	88.9	29.4	103.0	19.0	101.0
100 000-lb. Olsen	0.05	11.3	83.3	13.8	96.4	27.0	76.2	20.3	95.6	16.7	97.4
	1.04	13.4	84.8	9.1	87.5	23.0	85.2	20.7	95.6	15.9	94.3
50 000-lb. Riehlé	0.05	15.3	83.4	11.2	78.7	40.9	82.4	28.3	84.4	21.5	82.9
	0.78	15.3	100.0	15.5	97.2	32.6	93.5	31.1	110.0	23.6	85.0
SPECIMENS HELD IN SHOULDERED GRIPS, 2-in. Gage Length											
100 000-lb. Riehlé	0.10	5.33	93.3	5.50	74.3	9.79	70.8	10.40	85.9	7.67	88.9
	0.61	2.71	70.1	4.80	75.5	8.02	68.5	9.96	93.4	6.96	89.0
100 000-lb. Olsen	0.05	3.42	86.6	4.87	70.4	8.16	70.4	6.42	97.2	6.96	90.2
	1.04	3.84	68.0	4.95	57.3	6.53	61.7	6.81	74.7	6.07	93.6
50 000-lb. Riehlé	0.05	2.90	73.6	3.93	88.4	11.60	60.6	15.40	100.0	7.63	88.1
	0.78	2.42	93.3	3.63	87.0	12.60	57.0	10.30	101.0	9.77	96.1

[a] The term "elastic" applies to the range of load from about one-third the yield strength to yield strength. The term "plastic" applies to the range of load in the region of "ultimate" (maximum) load.

A series of tests was run in the Talbot

Laboratory at the University of Illinois to study the relation between speed of head of screw-power testing machines and rate of strain in a tension test specimen. The following variables were studied:

Testing Machine.—(1) a 100,000-lb. 2-screw Riehlé machine which has been in service about 35 yr.; (2) a 100,000-lb. 3-screw Olsen machine which has been in service about 19 yr.; and (3) a 50,000 Riehlé 2-screw machine which has been in service about 28 yr.

Grips.—For each machine tests were made with wedge grips and with shouldered grips.

Length of Specimen.—Tests were made on specimens with 8-in. gage length and on specimens with 2-in. gage length.

Metals.—S.A.E. 1020 steel, S.A.E. 1045 steel, duralumin (17 S-T), hard-drawn brass, hard-drawn copper.

Speeds of Head.—The slowest speed and the highest pulling speed were used on each machine.

The rate of strain was measured by the same resistance extensometer and oscillograph combination described in a previous paragraph. A similar device was used to measure actual speed of head as distinguished from nominal, but for these machines the ratio of actual and nominal speeds of head for any given metal was found to be nearly constant. During the "plastic stage" (yield to fracture) the ratio is very nearly unity.

Table III shows the ratios of rate of specimen stretch in the gage length to rate of nominal speed of head for the various combinations of testing machines, speeds of head, gripping devices, gage lengths of specimen, and different metals. These ratios are expressed in per cent.

A study of this table will show that the ratio of rate of specimen stretch to speed of head varies with different machines, different grips in a machine, different gage lengths of specimen, and different metals. For the two speeds used with any machine, the ratio, in general, is fairly constant, with some distinct exceptions, especially for brass specimens tested to the ultimate strength using wedge grips.[4] This means that while rate of strain cannot be predicted directly from speed of head of testing machine, if the ratio between rate of strain and speed of head is experimentally established for one speed of head it can be predicted with fair accuracy for any other speed of head at which the weighing apparatus of the machine will behave properly. A further study of the constancy of the ratio of rate of specimen stretch to speed of head is given in an appendix to this paper.[5]

Proposed Methods of Determining Speed to Be Used for Tension Tests:

Machines with Automatic Control of Rate of Strain or Rate of Loading.—Several makers of testing machines now have on the market machines fitted with automatic devices for control of rate of strain, rate of loading, or speed of head to any desired value within a considerable range. The problem of testing at proper rates of strain is much simplified when such devices are available. The authors have not had sufficient experience with them on which to base a reliable judgment of their accuracy, ruggedness, and service under severe laboratory conditions. It seems probable that they will come into wider use in the future. The remainder of this discussion will concern itself with testing machines not fitted with such automatic controls.

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[4] One conclusion which the authors drew from these tests was that hard-drawn brass rod should not be tested using wedge grips. The ends of the specimen are mashed, erratic slipping takes place, and the specimen frequently slips out of the grips or breaks near one of the grips outside the gage line.

[5] See p. 623.

Speed of Testing Up to Yield Strength.—It is usually convenient to take advantage of the approximate constancy of ratio between stress and strain up to yield strength. The rate of loading can then be used as a measure of rate of strain, using the equation:

$$R = \frac{100P}{AEt}$$

where:

R = rate of strain in per cent per minute,
P = change of load in pounds for a period of t minutes,[6]
A = area of cross-section of specimen in square inches, and
E = modulus of elasticity of material in pounds per square inch.

Consider first the screw-power machine with a balance beam for weighing load. A specimen of the lot to be tested is placed in the grips, the machine run at its lowest speed of head, and with a stop watch, the interval measured from the time the load reaches a value corresponding to (say) one-third the estimated yield strength to the time when the load reaches a value (say) two-thirds the estimated yield strength. From this measurement, assuming a constant ratio of head speed to rate of strain, R may be computed and the speed of head adjusted to give a rate of strain within the limits which may be set for the material to be tested. This method is essentially the same as that proposed by Withey in his report on the yield point of structural steel.[7]

Consider next the machine, either hydraulic or screw power, with a self-indicating load-weighing mechanism. Many such machines are equipped with a timing dial, which moves a pointer, a spot of light, or a disk with dots on it at a controllable rate of speed. The dial is set in motion at the rate of loading which corresponds to the desired value of R, and the gears of the screw-power machine are set to give the motion of the load-indicating pointer as nearly as possible the same velocity as the timing disk. If a hydraulic machine is used the speed is adjusted by manipulating the valves until the motion of the load-indicating pointer synchronizes with that of the timing disk.

If the machine is not equipped with a timing disk the method outlined for the screw-power machine with a balance beam may be followed. The load is then read directly from the load-indicating dial.

Speed of Testing from Yield Strength to Fracture.—The effect of speed of testing upon tensile properties is, in general, less above the yield strength than below it, except for the stainless steel and the cast metals with relatively low ductility. However speed of testing does have some effect especially on the elongation after fracture. Methods of making tests in that region within a given range of rates of strain seem worthy of consideration.

Consider first tests with a screw-power testing machine. The time which elapses between yield strength and fracture may be measured by means of a watch, using a speed of head which, it is estimated, gives approximately the desired rate of strain. From the elongation of the specimen after fracture and the time noted from yield strength to fracture the average rate of strain in the plastic range of strain may be determined with a fair degree of accuracy. The rate of strain corresponding to each available speed of head of testing machine may then be computed on the assumption that rate of strain is proportional to speed of head. Then that speed of head can be selected which will give most closely the desired rate of strain.

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[6] At the beginning of a tension test on a screw-power machine the rate of loading is not constant, but increases with the load. As the load increases, the rate of loading approaches uniformity. In the tests herein reported, except the tests of cast metal, soft-temper brass, and hot-rolled copper for a given speed of head, the rate of loading became practically constant above a load one-third to one-half of the yield strength load.

[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 139 (1931); Vol. 37, Part I, p. 87 (1937).

Consider next tests with a hydraulic testing machine. In most hydraulic machines one or more of the valve wheels is graduated so that duplicate settings can be made. After the oil in the testing machine cylinder has been warmed up by running the head up and down, or by making a few dummy tests, there is usually a fairly good correlation between setting of valve wheel and speed of head of testing machine. This correlation may well be checked for one or two settings before starting a series of tests. Using the valve setting to determine the speed of head, the rate of strain in the plastic range for any speed of head may be determined as in the case of the screw-power machine. Then the speed of head to give the desired rate of strain may be computed and the proper valve setting for the test determined.

APPENDIX

VARIATION FROM THE MEAN VALUE OF THE RATIO OF RATE OF STRETCH TO SPEED OF HEAD IN TESTS AT DIFFERENT SPEEDS AND IN DUPLICATE TESTS

In this further study of the ratio between rate of stretch of specimen and speed of head of testing machine the following symbols will be used:

R = the ratio of rate of stretch of specimen to nominal speed of head of testing machine in inches per minute,

R_f = the above ratio for the fastest speed used with a given testing machine, and

R_s = the above ratio for the slowest speed used with the same testing machine.

Taking all the values of R_s and R_f given in Table III, the average value of R_f/R_s is found to be 1.02 or 102 per cent. However, there are wide variations from this mean value in the case of individual tests.

It is felt that these variations from mean value are not to be attributed wholly to the varying speeds of testing for different tests. Light is thrown on this point by a comparison of the average deviation from the mean value of R for two arrangements of the test data: (1) An arrangement in which is studied the average deviation of average values of two tests for each of two speeds for each testing machine (data given in Table III), and (2) an arrangement in which is studied the deviation from the mean of the value of R for each of the two specimens in each pair of duplicate tests made (data not given in Table III, only the average for each duplicate pair of tests, data on file at the Talbot Laboratory).

For the two speeds for each machine the upper part of Table IV shows the average values of deviation R from the mean value for different metals, different gage lengths and different grips. The general average of this deviation is 6.86 per cent.

The lower part of Table IV shows the deviation of R for duplicate tests of specimens with speed, gage length, machine, and metal the same for each specimen of a pair. The general average of this deviation from the mean is 8.03 per cent.

Now it seems to the authors that these figures indicate that the effect of varying speed on variation of values of R was not greater than the effect of variation of metal in the duplicate specimens of a pair.

In connection with Table IV attention is called once more to Table II. From the values in Table II it is seen that even if an error as high as the greatest deviation in Table IV (23.2 per cent) were introduced into the rate of strain, the effect on any of the mechanical properties of any of the metals tested would be less than 1.65 per cent (except for the rather uncertain small reduction of area for the cast aluminum alloys and the cast magnesium alloy). This figure is based on the approximate proportionality of change in mechanical property value to the logarithm of the change in rate of strain.

TABLE IV.—AVERAGE FOR THREE TESTING MACHINES OF THE DEVIATION FROM THE MEAN VALUE OF *R*.

Values are given in per cent.

Grips	Gage Length, in.	Metal									
		S.A.E. 1020 Steel		S.A.E. 1045 Steel		Duralumin		Brass[a]		Copper	
		Elastic Range	Plastic Range	Elastic Range	Plastic Range	Elastic Range	Plastic Range	Elastic Range	Plastic Range	Elastic Range	Plastic Range
AVERAGE OF TWO TESTS FOR TWO SPEEDS (computed from data in Table III).											
Wedge	8	3.26	4.54	1.19	0.34	0.55	12.96	....	....	3.72	18.59
Wedge	2	2.69	7.14	0.68	4.79	5.66	13.50	....	....	7.03	17.84
Shouldered	8	6.09	4.72	15.40	5.88	8.83	5.60	....	....	6.73	3.72
Shouldered	2	15.80	12.70	3.86	3.93	8.39	3.76	....	....	8.00	1.99
Average		6.97	7.28	5.28	3.72	5.85	8.95	....	....	6.38	10.50
General Average, 6.86											
DEVIATION FROM MEAN FOR PAIRS OF DUPLICATE TESTS.											
Wedge	8	3.06	2.12	2.53	6.26	3.54	10.00	....	....	2.87	20.70
Wedge	2	4.90	6.47	3.66	8.43	5.69	5.59	....	....	5.93	23.20
Shouldered	8	7.77	7.05	8.74	2.72	8.91	7.29	....	....	14.20	3.80
Shouldered	2	20.60	5.32	13.50	14.20	7.08	8.86	....	....	7.25	4.66
Average		9.08	5.24	7.08	7.90	6.30	7.92	....	....	7.58	13.10
General Average, 8.03											

[a] The tests of hard-drawn brass rod held in wedge grips showed so much slipping in grips and mashing of ends of specimen that the results of all tests on brass rod have been omitted from this table, since to include some values for brass and exclude others would unbalance the general average.

[See joint discussion on Effect of Speed of Testing, p. 637.—Ed.]

SPEED IN TENSION TESTING AND ITS INFLUENCE ON YIELD POINT VALUES

By Lawford H. Fry[1]

Synopsis

The results of over 300 tension tests made at various speeds and on a number of testing machines are studied to observe how the rate of pulling affects the value found for the yield point. Some attention is also given to the effect of speed on the value found for the tensile strength.

It is shown that within the range of pulling speeds normally available, the influence of speed on the value of the tensile strength is practically negligible. The influence of the rate of pulling on the yield point is much greater. The figures indicate that with a tenfold increase in the rate of straining, the increase in the value found for the yield point will be about 7000 psi. in a 2-in. gage length specimen and about 2500 psi. in specimens with 8-in. gage lengths.

Even with careful and uniform testing practice, there is considerable scatter in the value found for the yield point of specimens from the same material. A scatter of 3000 psi. about a mean yield point value of 47,000 psi. is not unusual. Deviations are likely to fall below the true yield point value, rather than above.

In view of the dependence of the value of the yield point on the rate at which the test is pulled, limitation of testing machine speed is a proper requirement in acceptance specifications. The test data show that with constant crosshead speed, the rate of load application, below the yield point, varies as the load is applied. For use in calibrating screw-driven testing machines the idea of the mechanical modulus of the machine is developed. This connects the rate of loading or rate of straining of the specimen at any load with the rate of crosshead travel. By definition, the modulus is the instantaneous rate of change of the load in pounds of load added per inch of crosshead travel. The value of the modulus varies with the load, but for any given load is independent of the crosshead speed.

Many of the acceptance specifications under the jurisdiction of the Society's Committee A-1 on Steel call for determination of the yield point. In this paper, the results obtained in the routine testing used in the application of these specifications are studied. Attention is directed only to those materials which have a true yield point. This applies particularly to the milder structural steels and to wrought iron. As the late J. A. Capp pointed out, nature has been kind to the man who tests these materials. Their stress-strain curve shows a well-marked jog just beyond the nominally straight-line portion of the curve. As a result of this juxtaposition, the determination of the jog, or yield point, in routine testing furnishes a convenient check on a property of the material which is useful to the design engineer. The jog marks the Yield Point, which is defined as the stress at which there occurs a sudden increase of strain without corresponding increase in stress.

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Long after determination of the yield point had become standard procedure in tension testing, testing engineers were confronted with the problem of handling newer alloy steels and non-ferrous metals whose stress-strain curves have no jog. The curves bend over smoothly and may or may not show an initial straight line. An example of the sharp-kneed curve with a jog marking the yield point is shown in Fig. 1, together with an example of the smooth continuous curve. The testing engineer, with the example of the sharp-kneed materials before him, has endeavored to repair nature's neglect of the smooth curve materials by inventing the so-called Yield Strength, with values set up on an arbitrary basis. The Yield Strength may serve a purpose similar to that of the Yield Point, but the two are entirely distinct. Confusion would have been avoided if the sponsor of Yield Strength had recognized that no real yield is involved and had not infringed so closely on the old established name. Our concern here is with the Yield Point, a physical characteristic of the sharp-kneed materials, and not with the arbitrary and artificial Yield Strength.

It is known that the value found for the yield point of a material depends on the speed at which the test specimen is pulled. Study of the relation between the two involves consideration of two factors: (1) the relation which the rate of straining of the specimen bears to the value found for the yield point, and (2) the effects which design of testing machine and type of test specimen have on the rate of straining produced by a given crosshead speed. At a given crosshead speed two different machines with the same test specimen, or the same machine with two different types of specimen, may give entirely different rates of straining with the same crosshead speed. The data available for the paper show the relation between rate of straining and yield point values as found in over 200 individual determinations made on 30 testing machines of various types operated under widely varying conditions.

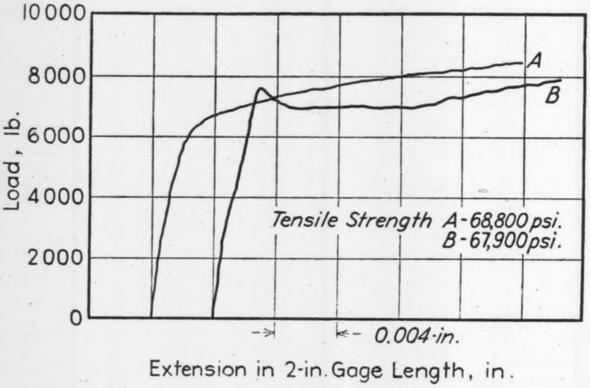

FIG. 1.—Comparison of Sharp-Kneed and Smooth Stress-Strain Curves.
Specimen 0.505 in. in diameter by 2-in. gage length.

Scatter in Yield Point Value:

Before considering the data as a whole, it is desirable to consider the degree of precision secured in the measurement of the yield point under normal conditions of routine testing.

In Fig. 2 are plotted three series of tests made with the same material. The specimens were standard ½-in. diameter, 2-in. gage length specimens. The tests were made by careful observers under conditions which were uniform except for the variations in speed. The tests of series X and Y were made by a single observer using a different machine for each series. At each speed six specimens were pulled in series X and three in series Y. The plot shows by the length of the vertical line the range of values observed at each speed. The mean value is also marked. The third series of tests, marked SL, SE and SD, is made up of results reported by three observers, each using his individual machine. On each machine three specimens were pulled at a single speed. The three machines were each run at a different speed.

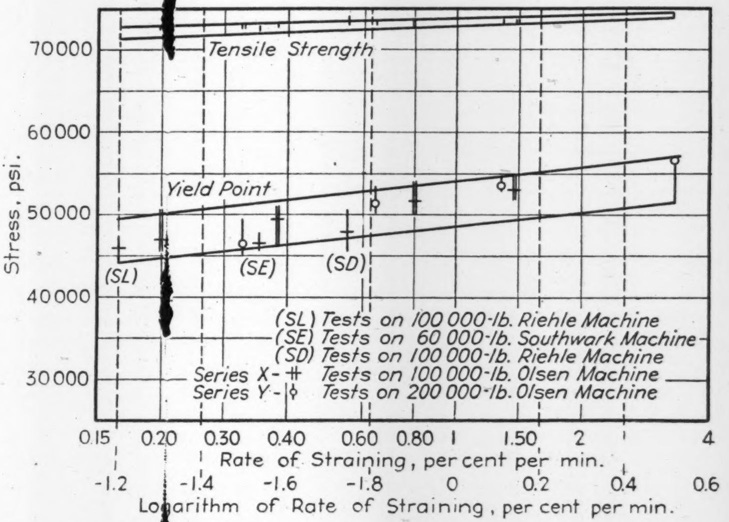

FIG. 2.—Relative Spread of Yield Point and Tensile Strength Determinations.

Values for the tensile strengths are also plotted for each series. It is at once evident that with uniform material tested by a single observer under exactly similar conditions, the value of the tensile strength is much more precise than that of the yield point. With the same machine and the same speed the yield point values may have a scatter of 3000 psi. about a mean value of 47,000 psi. For the tensile strengths the scatter is only about 600 psi. about a mean value of about 72,000 psi.

Undoubtedly the greater scatter of the yield point values is due to the nature of the yield. As the specimen reaches its ultimate strength, it is stretching plastically and uniformly. The stress is measured under conditions which are essentially stable. The yield point, however, is measured under entirely different conditions. The metal is strained elastically at first, but as the yield point is approached the elastic condition becomes unstable. At some

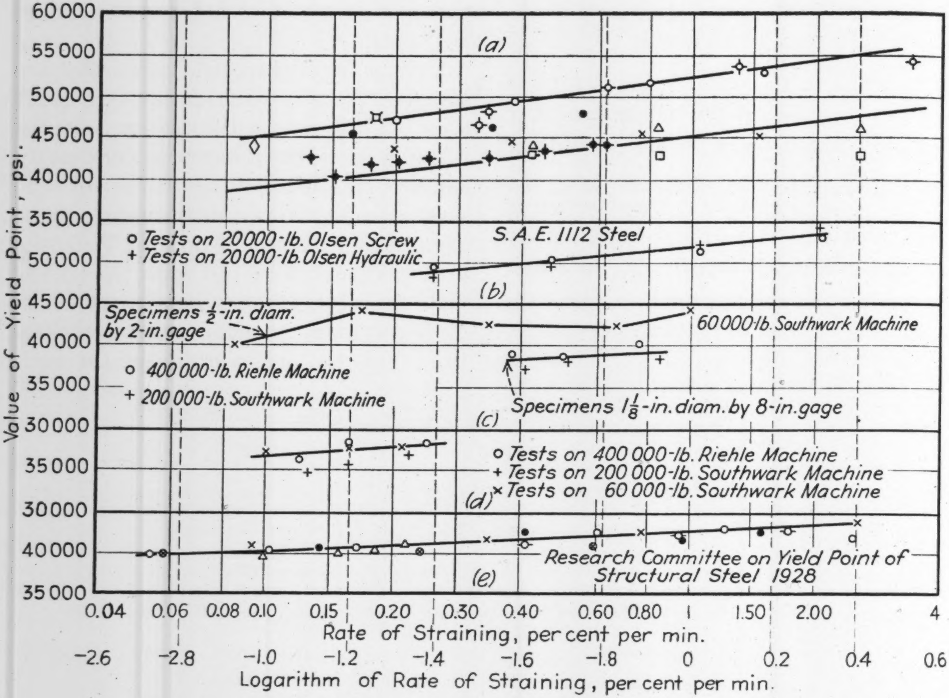

FIG. 3.—Relation Between Yield Point Values and Rate of Straining.

(*a*) All tests made with ½-in. diameter by 2-in. gage length test specimens from Bethlehem Heat 5 J 701.

Test Series:

○ 100,000-lb. 3-screw Olsen machine. Four crosshead speeds, 1/16 to ½ in. per min., 6 specimens at each speed.

Total number of tests........................ 24

✦ 200,000-lb. 4-screw Olsen machine. Four crosshead speeds, 1/16 to ½ in. per min., 3 tests at each speed.

Total number of tests........................ 12

× 60,000-lb. and 200,000-lb. Southwark-Emery machines. Four pacing-dial speeds 11,700 to 86,600 lb. per min. Two specimens on the 60,000-lb. and four on the 200,000-lb. machine at each speed.

Total number of tests........................ 24

△ 300,000-lb. Southwark-Emery machine. Three pacing-dial speeds, 25,000, 50,000, and 75,000 lb. per min. Six specimens at each speed.

Total number of tests........................ 18

Results reported by Metallurgical Committee of Steel Founders' Society of America. Sixteen machines screw driven and hydraulic, of capacities from 30,000 to 100,000 lb. Each machine pulled 3 specimens at a single speed.

⌑◇ Special test specimen holders.

✦ Shouldered test specimens.

● Threaded test specimens.

Crosshead speeds ranged from 0.045 to 0.125 in. per min., producing rates of strain 0.095 to 0.554 per cent per min.

Total number of tests........................ 48

Grand Total Number of Tests Fig. 3 (*a*)........ 126

(*b*) Specimens ½-in. diameter by 2-in. gage length of S.A.E. 1112 steel. 20,000-lb. hydraulic loading lever weighing, pendulum indicating and 20,000-lb. gear box drive, four screw, dial Vernier scale beam Olsen machines were used. Four speeds, 1/16 to ½ in. per min., loading rates 14,760 to 121,500 lb. per min. Four specimens, each machine each speed.

Total number of tests........................ 32

(*c*) Specimens, 1⅛-in. diameter bars as rolled, 8-in. gage length. 400,000-lb. Riehle screw-driven machine, three crosshead speeds, 0.280, 0.367, and 0.560 in. per min., three specimens at first and two at each of other speeds.

Total number of tests........................ 7

200,000-lb. Southwark-Emery machine, pacing-dial speeds 12,000, 15,000, and 25,000 lb. per min., two specimens each speed.

Total number of tests........................ 6

Specimens ½-in. diameter by 2 in. gage length turned from 1⅛-in. bars, pulled in 60,000-lb. Southwark-Emery machine at five pacing-dial speeds 5000 to 60,000 lb. per min.

Total number of tests........................ 9

(*d*) Specimens ⅜ by 1¾ in. with 8-in. gage length. 400,000-lb. Riehle screw-driven machine, crosshead speeds 0.280, 0.367, and 0.560 in. per min., two specimens each speed.

200,000-lb. Southwark-Emery machine, pacing-dial speeds 24,000, 30,000, and 42,000 lb. per min., two specimens pulled at each speed.

60,000-lb. Southwark-Emery machine, pacing-dial speeds 30,000, 45,000, and 60,000 lb. per min., eight specimens pulled at each speed.

Total number of tests........................ 36

(*e*) All specimens were ¾-in. diameter bars as rolled with 8-in. gage length. One test series was pulled on a Southwark-Emery 60,000-lb. machine at four pacing-dial speeds, 30,000 to 65,000 lb. per min., three to five tests at each speed.

Total number of tests........................ 17

The other five series are from test data reported by the Research Committee on the Yield Point of Structural Steel in 1928, *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 110. Five laboratories cooperated, using six screw-driven testing machines of capacities from 60,000 to 400,000 lb. Each laboratory pulled five specimens at each of four crosshead speeds ranging from 0.05 to 2.0 in. per min. All specimens were from the same heat of steel.

Total number of tests........................ 100

Grand Total Number of Tests Fig. 3........... 333

point the elastic equilibrium is disturbed and yielding begins. This does not occur simultaneously throughout the specimen. It starts at some individual point at which the stress first reaches the value of the true yield point. As the metal at this point yields, it stretches rapidly, transferring its load to the surrounding metal, which in turn yields. This continues until the total elongation is sufficient to let the beam of the machine drop. At this time the testing machine measures the average stress in the specimen at which the beam drops. This must always be less than the concentrated stress at which the true yield point was reached and yielding began. If a specimen were perfectly straight and if the holders transmitted a truly axial load without causing any stress concentration, the testing machine would measure the true value of the yield point. Any stress concentration due to a bent specimen or to eccentric loading or to uneven bearing of the holders will start local yielding before the average stress has reached the true yield point value.

The true yield point can be found only if special precautions are taken. Any deviations from perfection in specimen or in the supporting devices will tend to give too low a value for the yield point. With reasonable accuracy in measurement the observed value will be too low rather than too high. This is well illustrated by the experimental data reported by Morrisson.[2] Yield point determinations were made under carefully controlled conditions with three extensometers on each specimen. The specimens were machined with a high degree of accuracy, and special precautions were taken to secure true axial loading. In spite of this, the extensometer readings showed that the observed yield point stress was considerably lower than the maximum stress at which the yield point was reached. This is shown in the following tabulation:

Yield Point Values, psi.		Correction Added	
Observed	Corrected	psi.	per cent
49 460	52 270	2810	5.9
48 160	51 590	3430	7.1
49 750	52 080	2330	4.5

With this in mind, it seems reasonable to believe that in routine testing the measured yield point values fall below rather than above the true value, and that the scatter observed is due to variations in the stress distribution as applied to the individual specimens.

In presenting the remainder of the experimental data, mean yield point values are given, and in considering trends it must be remembered that each point plotted represents the mean of several tests with a considerable amount of scatter.

Yield Point Value versus Rate of Straining:

In Fig. 3 the mean yield point values from Fig. 2 are replotted, together with the mean values from a number of other tests. The upper section, Fig. 3 (*a*), presents the results obtained by a number of observers using various types and sizes of testing machines. All test specimens were ½-in. diameter by 2-in. gage length and were taken from a single heat of steel, Bethlehem 5 J 701. The specimens were machined from ¾-in. diameter rolled bars which had been stress relieved.

Mean yield point values are plotted as ordinates on abscissae which represent rates of straining in percentage per minute plotted on a logarithmic scale. Rates of straining range from 0.093 to 3.3 per cent per minute. With the ½-in. by 2-in. specimens these rates of strain correspond to loading rates ranging from

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[2] J. L. M. Morrisson, *Proceedings*, Inst. Mechanical Engrs., January, 1940.

28,000 to 1,000,000 psi. per minute, or from 5600 to 200,000 lb. per minute. These rates extend beyond normal testing speeds in both directions, particularly at the upper end.

It is evident that there is a general tendency for yield point value to rise as the rate of straining is increased. The amount of rise is indicated by the slope of the lines which include the majority of the tests. This slope corresponds to an increase of about 7000 psi. for a tenfold increase in rate of straining.

In Fig. 3 (*b*) results for another series of tests with $\frac{1}{2}$-in. by 2-in. specimens are plotted. These were made by one observer using two 20,000-lb. capacity machines, one screw driven and one hydraulically operated. Four specimens were pulled at each speed, all being taken from an S.A.E. 1112 steel having very nearly the same yield point as the 5 J 701 steel of Fig. 3 (*a*). Mean yield point values are plotted and show closely similar results for both machines. The rise in yield point value with increase in rate of strain is very nearly the same as that noted above for the other $\frac{1}{2}$ by 2-in. specimens.

The three lower sections of Fig. 3 plot yield point values obtained with test specimens of various dimensions taken from various steels. Figure 3 (*c*) presents results from specimens $1\frac{1}{8}$ in. in diameter as rolled, by 8-in. gage length, and from $\frac{1}{2}$-in. diameter by 2-in. gage length specimens turned from the $1\frac{1}{8}$-in. bars. Figure 3 (*d*) presents results from specimens $\frac{3}{8}$ by $1\frac{3}{4}$ in. with 8-in. gage length pulled on three different machines. Figure 3 (*e*) presents the results reported in 1928 by the Research Committee on the Yield Point of Structural Steel. Specimens were bars $\frac{3}{4}$ in. in diameter as rolled, with 8-in. gage length, all taken from one heat of steel. It also includes one set of similar specimens from another heat of steel tested recently. Of the three groups of tests with 8-in. gage length specimens, Fig. 3 (*e*) gives the clearest relation between yield point value and rate of strain. The line shown gives an increase of about 2500 psi. for a ten-fold increase in rate of straining. The points in Fig. 3 (*c*) and (*d*) do not give a precise relation, but the lines which have been drawn parallel to that of (*e*) do not conflict with the individual results.

Comparison of Fig. 3 (*a*) and (*e*) suggests that with the 8-in. gage length the rise of yield point value with increase in speed is less rapid than with the 2-in. gage length. This fits in with practical experience and is believed to be true. The tests in Fig. 3 (*c*) with 2-in. specimens turned from the $1\frac{1}{8}$-in. bars were planned to throw light on this question, but the results are not precise enough to serve as confirmation or contradiction.

Measurement of Rate of Straining:

In the preceding section, yield point values have been compared on the basis of rates of straining, without considering how these rates are or should be measured. In the case of the hydraulically operated machines with pacing dial, there is no problem. The dial is set for a desired rate of loading and the indicator is kept at pace with the dial by the appropriate adjustment of the control valve. With a screw-driven machine, complications enter. Control of speed is limited to the choice of one of a limited number of gear settings, each of which gives a corresponding uniform rate of crosshead speed. The rate of loading or rate of straining produced by each speed depends on the design of the testing machine and the type of test specimen used.

Even with machines using the same type of specimen, wide variations in rate of straining may be produced by the same crosshead speed. This is illustrated by the following tabulation, which is based on data reported by the Metallurgical Committee of the Steel Founders Society of America:

Laboratory	Machine	Yield Point, psi.	Average Rate of Loading Between 1000 lb. and 7500 lb., lb. per min.	Rate of Straining of Specimen, per cent per min.	Mechanical Modulus of Machine, lb. increase in load per inch of free crosshead travel
SB	Riehle—100 000-lb.	41 630	10 600	0.176	85 000
SC	Riehle—100 000-lb.	42 520	20 200	0.336	162 000
SH	Riehle—100 000-lb.	43 180	27 000	0.450	216 000
SG	Southwark—100 000-lb.	44 000	35 500	0.591	284 000
SN	Riehle— 50 000-lb.	45 050	32 500	0.544	260 000

Yield point determinations were made by five different laboratories all using a free running crosshead speed of 0.125 in. per min. with standard ½-in. diameter by 2-in. gage length specimens. The time required for the load to rise from 1000 to 7500 lb. was noted. From this the mean rate of loading and mean rate of straining have been computed. The fourth column shows that for the same crosshead speed, the range of the rates of straining of the different machines is in the proportion of 1 to 3. It will also be seen that the value found for the yield point rises as the rate of straining increases, although the crosshead speed is the same in all cases. From this it is evident that control of speed of testing in the interest of uniform yield point values must be based on rate of straining rather than on crosshead speed.

In the tests tabulated above, the rate of straining has been measured by timing the increase of load through a given range. This would give satisfactory results if the rate of straining were uniform throughout the test. Unfortunately the rate of straining, even with a uniform rate of crosshead travel, varies considerably as load is added during the test. Therefore, measurement of an average rate of loading for a haphazard range of loads does not provide information as to the rate of loading or straining at the yield point load. For this purpose a calibration curve is needed showing the variations in rate of loading throughout the whole loading range.

Such calibration curves can be based conveniently on a relation that it is proposed to call the mechanical modulus of the testing machine. The value of this modulus depends on the whole test setup, machine, grips and specimen. With a given setup the value of the modulus varies with the load, but at any given load it is the load in pounds added per inch of crosshead travel. As an aid to understanding the meaning of the modulus, consider the mechanism by which the movement of the crosshead applies load on the specimen. The full travel of the crosshead does not appear directly as strain in the specimen. The whole system of machine, grips, and specimen is strained and only a small fraction of the total strain appears as strain in the specimen. The setup of machine grips and specimen may be compared to a strained spring in which a load is produced by a movement or deflection of one end. The properties of such a spring are measured by the spring characteristic, that is, the load added per inch of deflection. The modulus suggested corresponds to the spring characteristic of the setup. It is expressed as pounds of load added per inch of crosshead travel. In a simple spring the spring characteristic is constant for all deflections. In the testing machine setup this is not the case. The value of the modulus, that is, the pounds of load added per inch of crosshead travel, varies with the load, as shown by the curves in Fig. 4. Although the value of the modulus varies with the load, it is, generally speaking, independent of the rate of crosshead travel. At any load the value of the modulus is fixed and the pounds of load added per inch of crosshead travel is the same for all crosshead speeds. In other words, at any given load the rate of increase in load is directly proportional to the rate of crosshead travel. Therefore, if modulus values are plotted against load as in Fig. 4, the resulting curves can be used to find at once for any load the rate of straining which will be produced by any rate of crosshead travel. The curves in Fig. 4 have been developed by the methods described in the Appendix.[3] They apply to a variety of combinations of testing machine and test specimens and vary greatly in form from one combination to another. The application of the curves to the determination of the rate of straining at the yield point is extremely simple. Take, for example, curve *C* of Fig. 4. For a yield point of 45,000 psi., a load of 9000 lb. on the ½-in. specimen, curve *C* shows a modulus of 172,000 lb. per in. of crosshead travel.

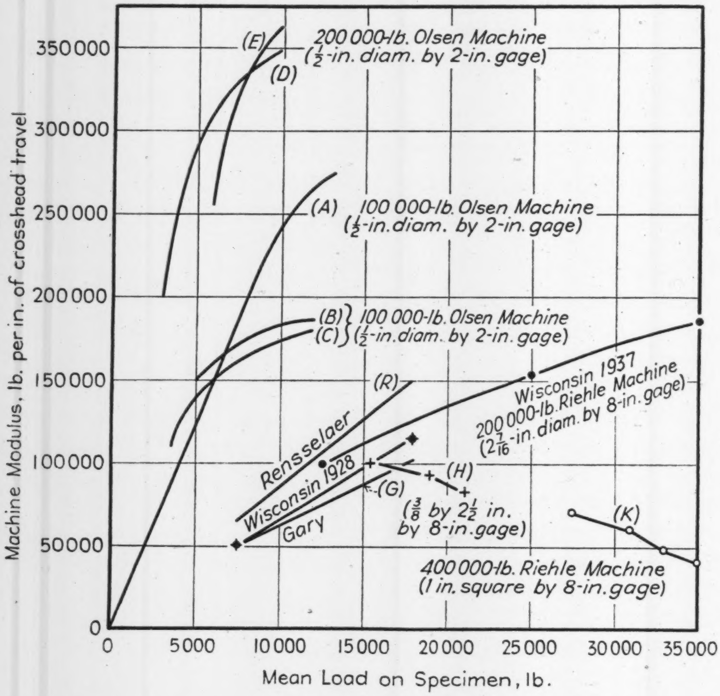

FIG. 4.—Testing Machine Modulus in Relation to Load.

If the machine is geared to give crosshead speeds of 1/16, ⅛, ¼, and ½ in. per min., the rates of loading, stressing, and straining will be:

Crosshead Speed, in. per min.	Rate of Loading, lb. per min.	Rate of Stressing, psi. per min.	Rate of Straining, per cent per min.
1/16	10 750	53 750	0.18
⅛	21 500	107 500	0.36
¼	43 000	215 000	0.72
½	86 000	430 000	1.44

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[3] See p. 634.

For a machine with a calibration curve as curve D in Fig. 4, the modulus at a load of 9000 lb. has a value of 344,000 lb. per in. of crosshead travel and the rates of loading, etc., will be just double those given above for the same crosshead speeds.

GENERAL CONCLUSIONS

Study of the experimental data leads to the following conclusions:

1. Yield point values determined under normal conditions of routine testing show a scatter considerably greater than can be attributed to variations in the quality of the material. Such variations are undoubtedly due to variations in stress concentration during testing, so that observed values fall below rather than above the true yield point value.

2. Yield point values tend to increase with an increase in the rate at which the test specimen is pulled. This increase is related directly to the rate of straining and only indirectly to the rate of crosshead travel. No systematic difference is found between yield point values determined on screw-driven and hydraulically operated testing machines.

3. Yield point value increases generally in a straight-line relation with the logarithm of the rate of straining. The increase in value with a three-fold increase in the rate of straining is of about the same order as the scatter between values obtained from specimens of the same material. Consequently, an extremely precise control of rate of straining is not warranted. There is some indication that for the same increase in rate of straining, the rise in yield point is less rapid with specimens with 8-in. gage length than it is with specimens with 2-in. gage length.

4. Only a small fraction of the crosshead travel appears as strain in the specimen. The proportion of strain to crosshead travel varies during a test as the load is increased, but is unaffected by the rate of crosshead travel. At a given load, the load added in pounds per inch of crosshead travel is the same for all crosshead speeds. This relation, pounds of load added per inch of crosshead travel, has been named the Mechanical Modulus of the testing machine. It varies greatly for different combinations of machine and test specimen. For any given combination, a plot of modulus against load gives direct information as to the rate of straining produced at any load by any given crosshead speed.

If a limit is to be set on the rate of straining to be allowed with screw-driven testing machines, it is recommended that a load-modulus curve be set up and that this be used to estimate the rate of straining produced at the yield point by the crosshead speeds available.

Acknowledgment:

Observers who cooperated in carrying out tests undertaken for the purpose of the present study were A. G. Spooner, Bethlehem Steel Co., who arranged for the supply of the bars of Heat 5 J 701; F. G. Tatnall and C. B. Marshall of the Baldwin-Southwark Corp.; R. B. Lewis, Chief Engineer, Tinius Olsen Testing Machine Co.; Ray McBrian, Engineer of Tests, Denver and Rio Grande Western Railroad Co.; P. G. McVetty, Mechanical Engineer, Westinghouse Electric and Manufacturing Co., Research Laboratory; and L. C. Martin, Chief Inspector, Homestead Works, Carnegie-Illinois Steel Corp. The assistance of the Metallurgical Committee of the Technical and Operating Group, Division No. 1, Steel Founders' Society of America, in furnishing extensive test data is gratefully acknowledged.

APPENDIX

Modulus of Testing Machine

Study of yield point determinations made under widely varying conditions shows that the yield point value is directly affected by the rate of straining at which the specimen reaches the yield point. It is also found that this rate of straining is not only dependent on the rate of crosshead travel but is affected by the type of testing machine, by the type of specimen and the grips used, as well as by the load at which the yield point is reached. With the crosshead moving with a uniform speed, the load on the specimen is not applied at a uniform rate. The rate at which the load increases varies with the load on the specimen. Consequently, measurement of the rate of loading at one load does not by itself give the rate of loading or straining at which the yield point will be reached. For this purpose a calibration curve of the machine and specimen setup is needed

The load-modulus curves in Figs. 4 and 5 provide the necessary calibration data for the various combinations of testing machines and test specimens. The modulus of the testing machine is defined as the increase in load in pounds per inch of crosshead travel. The value of this modulus varies as the load on the specimen increases during the test, but, generally speaking, at any given instantaneous load on the specimen the rate of increase in load is directly proportional to the crosshead speed. In other words, at any instantaneous load the modulus is independent of the crosshead speed. For this reason a single load-modulus curve provides complete information as to the rate of loading for all crosshead speeds. As stresses are below the yield point, rate of straining is in direct proportion to rate of loading.

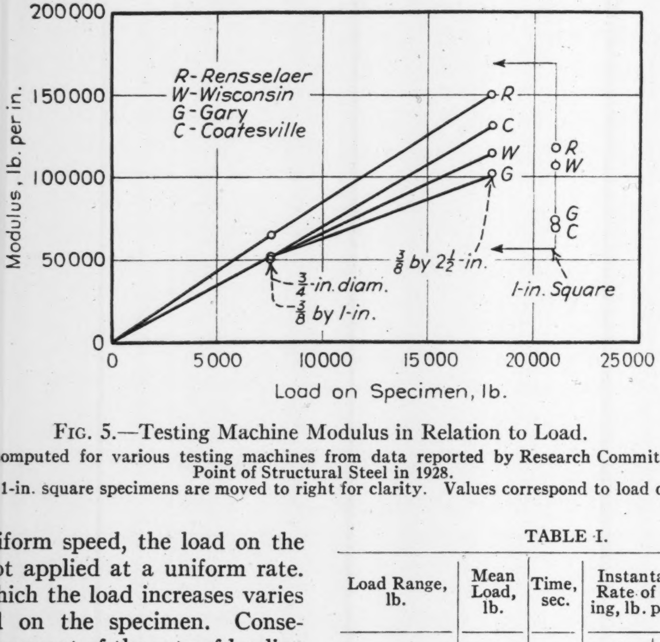

FIG. 5.—Testing Machine Modulus in Relation to Load.

Value of modulus computed for various testing machines from data reported by Research Committee on the Yield Point of Structural Steel in 1928.

Ordinates for 1-in. square specimens are moved to right for clarity. Values correspond to load of 18,000 lb.

TABLE I.

Load Range, lb.		Mean Load, lb.	Time, sec.	Instantaneous Rate of Loading, lb. per min.		Modulus, lb. per in.
w_1	w_2	w	t	$60\ dw/dt$	$60\ (w_1 - w_2)/t$	M
1000 to	6 000	3 500	93.6	4 000	3 200	83 500
2000 to	8 000	5 000	72.0	5 750	5 000	120 000
3000 to	9 000	6 000	63.9	6 200	5 600	129 000
4000 to	11 000	7 500	55.4	8 200	7 600	170 000
5000 to	12 000	8 500	48.8	9 600	8 600	200 000
6000 to	14 000	10 000	47.2	10 900	10 200	227 000
7000 to	14 000	10 500	35.4	12 400	11 900	258 000
8000 to	14 000	11 500	27.8	13 300	13 000	277 000

Figure 4 shows a number of load-modulus curves obtained from screw-driven testing machines with specimens of various types. Curves *A*, *B*, *C*, *D*, and *E* all apply to the A.S.T.M. standard test specimen of ½-in.

diameter by 2-in. gage length.[4] The rate of increase in load was measured by timing the rise in load over various load intervals. A typical series of observations and derived data for curve *A* are given in Table I. The measurements were made at a crosshead speed of 0.048 in. per min. The specimen was of high tensile strength steel so that it could be carried to loads of 14,000 lb., 70,000 psi., without yielding.

The values for each load were found by first determining the mean instantaneous rate of loading for that load. A typical set of observations for one of the tests is given in Table I. The first column shows the ranges over which the increase in loading was timed, while the second gives the mean load of this range. The third column gives the time for the loading range, and the fourth and fifth give computed instantaneous rates of loading for the mean load. Two methods of calculation are used. The values of the fourth column are computed on the assumption that the rate of loading is increasing with a straight-line relation throughout the loading range. The plotted results justify this assumption. As a matter of comparison, the fifth column gives the average loading rate obtained by dividing the increase in load by the time taken for this increase. The loading rates obtained by this method are slightly lower than those given in the fourth column, and with a straight-line plot as curve *A* (Fig. 4) are not so correct. If the curve drops so as to take a horizontal position, as curve *B* at the higher loads, the average values as in the fifth column of Table I will be more nearly correct.

It will be found that each combination of machine and specimen will give a definite and characteristic load-modulus curve, although individual measurements may not have a high degree of precision. In successive measurements there may be slight variations in motor speed or in the gripping of the specimen. Such variations, however, may occur in regular tests and are not sufficient to interfere with the usefulness of the load-modulus curve in measuring the rate of straining. In view of the fact that the variation in yield point value with a three-fold increase in rate of straining is of the same order as the scatter of the individual values at the same speed, it is evident that high precision in determination of the rate of straining is not necessary.

The Rensselaer and Gary curves of Fig. 4 are shown in greater detail in Fig. 5, which is based on data published by the Research Committee on the Yield Point of Structural Steel in their 1928 report. Four machines in as many laboratories were used, and with each machine rates of loading were measured at two different speeds. The rate of load increase was measured over two ranges, from 5000 to 10,000 lb., giving a mean load of 7500 lb., and from 12,000 to 24,000 lb., a mean load of 18,000 lb. For each load range two types of specimens were used. For the low range the specimens were respectively 1 by $\frac{3}{8}$ in. and $\frac{3}{4}$ in. in diameter. For the high load range, the specimens were respectively $2\frac{1}{2}$ by $\frac{3}{8}$ in. and 1 in. square. In all cases the gage length was 8 in.

Analysis of the experimental data shows that with all four types of specimens the crosshead speed has no practical influence on the value of the modulus, that is, on the load in pounds added per inch of crosshead travel. Also at the lower load range both types of specimen give similar results. At the higher load range a difference appears. The $\frac{3}{8}$ by $2\frac{1}{2}$-in. specimens, as shown in Fig. 5, have modulus values considerably higher than those for the 1 in. square specimens, except on the Wisconsin machine. In order to show this difference the abscissa over which the 1 in. square values are plotted has been displaced to the right. These points, as well as those for the $\frac{3}{8}$ by $2\frac{1}{2}$-in. specimens, should fall on a vertical line corresponding to a mean load of 18,000 lb. It is believed that the lower values for the square specimens are due to greater slippage in the grips. If flat wedge grips were used for both types of specimens, the flat $\frac{3}{8}$ by $2\frac{1}{2}$-in. specimens would be held more firmly, and for the same crosshead travel the load added would be greater than that added on the 1-in. square specimens.

It should be noted that the curves in Fig.

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[4] Standard Methods of Tension Testing of Metallic Materials (E 8 - 36), 1939 Book of A.S.T.M. Standards, Part I, p. 750.

5 are not carried far enough to serve as calibration curves at the yield point of the heavier specimens, but would serve for the smaller specimens. With a yield point of 44,000 psi., the load would be 11,750 lb. for the $\frac{3}{8}$ by 1-in. specimen and 10,000 lb. for the $\frac{3}{4}$-in. round. With the larger specimens the load at yield point would be 41,300 lb. for the $\frac{3}{8}$ by $2\frac{1}{2}$-in. specimen and 44,000 lb. for the 1-in. square specimen.

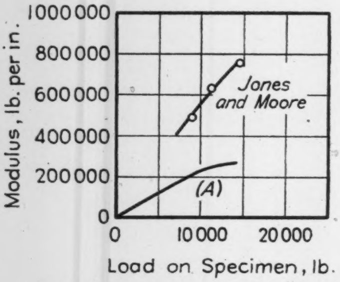

FIG. 6.—Modulus Values Computed from H. F. Moore's Test Data in Comparison with Curve *A* from Fig. 4.

Values for the Wisconsin machine are carried to much higher loads by the test data given in the 1937 report of the Research Committee on the Yield Point of Structural Steel. This curve, as shown in Fig. 4, was obtained with a specimen $2\frac{7}{16}$ in. in diameter. The curve differs little from an extension of the 1928 Wisconsin curve.

The two curves *H* and *K* obtained on the Riehle 400,000-lb.machine with $\frac{3}{8}$ by $2\frac{1}{2}$-in. and 1-in. square specimens reverse the trend and show a falling off in modulus values as the load on the specimen is increased. Both types of specimens were held in flat wedge grips, and it is believed that considerable slippage took place.

The paper by Jones and Moore[5] provides data from which modulus values for their machine can be computed. The tabulation which follows, gives the yield point and the corresponding rate of strain reported in Fig. 4 of their paper for a crosshead speed of 0.40 in. per min. for the three steels. It is to be noted that the higher the load at which the yield point is reached, the higher is the rate of strain for the same crosshead speed. Values computed for the modulus are plotted in Fig. 6. For comparison, curve *A* from Fig. 4 is repeated in Fig. 6.

Material	Yield Point		Rate of Strain, per cent per min.	Modulus, lb. per in.
	psi.	lb.		
S.A.E. 1020......	44 000	8 800	0.65	488 000
S.A.E. 1045......	54 500	10 900	0.85	638 000
Corten...........	70 500	14 100	1.05	756 000

It will be seen that for the same loads the modulus figures are much higher than any of those in Fig. 4. The modulus values for Moore's machine indicate a very stiff machine with a rapid increase in stiffness as the load increases.

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[5] See p. 610.

DISCUSSION ON EFFECT OF SPEED OF TESTING

Mr. H. W. Schmidt[1] *(presented in written form)*.—We are in agreement with Jones and Moore's conclusion that speed has no serious effect on the properties measured.

A few tests were made in The Dow Chemical Co. laboratory on the effect of speed of testing on the properties of magnesium alloys Dowmetal H and J. The tests were made on a 100,000-lb. screw powered testing machine. Means such as were employed by the authors were not available for accurate measurement of strain rate. The average rate of strain was calculated from the elongation after failure of the specimen and total elapsed time of the test. The ultimate tensile strength was determined up to those speeds at which the poise could follow the load increase with any degree of accuracy. No attempt was made to measure yield strengths. The accompanying Fig. 1 shows the effect of speed on the tensile strength, Fig. 2 the effect on the elongation, and Fig. 3 the effect on the reduction of area. In the case of Dowmetal H the effect on the properties was negligible in contrast to the decrease in properties observed by Jones and Moore.

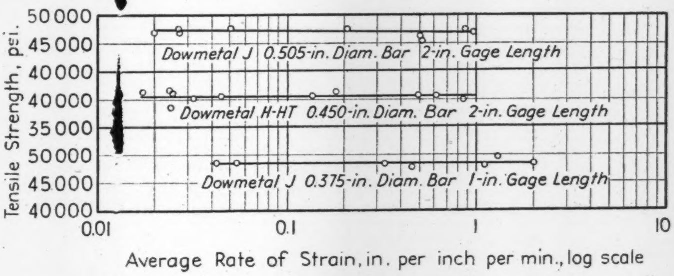

Fig. 1.—Effect of Speed on Tensile Strength.

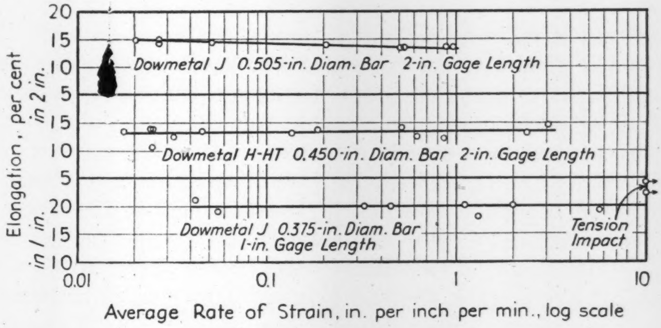

Fig. 2.—Effect of Speed on Elongation.

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[1] The Dow Chemical Co., Midland, Mich.

We believe that the change in properties as observed may be due to differences in bars as noted by Jones and Moore.

MR. R. H. CAREY[2] (*by letter*).—Messrs. Jones and Moore are to be congratulated on their very enlightening paper. This investigation is particularly interesting to the Department of Engineering Mechanics at The Pennsylvania State College. We are investigating, first, the effects of rate of straining; second, the relation between the dynamic constants of specimen and testing machine with respect to effect on the stress-strain diagram, especially the yield points.

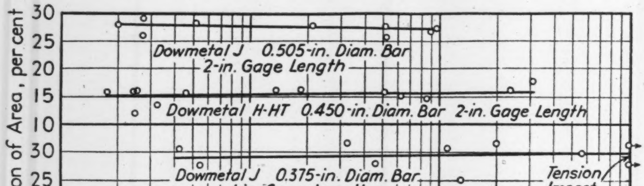

FIG. 3.—Effect of Speed on Reduction of Area.

This investigation is being carried out on equipment which is readily adaptable to any existing machines and does not require the use of an oscillograph. The machine used at present is a standard screw-driven machine, connected with a direct-current motor, the speed of which is controlled very accurately by means of a pacing disk. By means of this disk and two photocells, the rate of load application is controlled without appreciable inertia effect. A stress-strain recorder and Selsyn motor which drives an Ames dial in connection with the pacing arrangement, control the strain rate independent of any crosshead speed. This setup will be described in one of the forthcoming issues of the ASTM BULLETIN.[3]

MR. P. G. MCVETTY.[4]—In comparing these two papers we find that Mr. Fry used commercial testing procedure with a number of different machines while the investigation at the University of Illinois used a more precise method with one machine. Both methods have certain advantages and the fact that they reached similar conclusions adds to their value. Both have agreed that the significant factor which affects tension test results is the rate of strain rather than the crosshead speed. A large number of A.S.T.M. specifications now impose testing speed limitations in terms of crosshead speeds. It seems desirable to suggest that the committees responsible for these specifications consider a change to rate of strain or, in some cases, rate of application of stress.

Several factors affect the relation between crosshead speed and the strain rate communicated to the test specimen. In the region in which strain is proportional to load, Mr. Fry suggests that the combined effects of these factors for a particular machine and test specimen be expressed as a "testing machine modulus" in terms of pounds per inch of crosshead travel. When this modulus is multiplied by crosshead speed, we obtain the loading rate in pounds per minute. This is easily converted to strain rate, as shown by Jones and Moore, by dividing by the cross-sectional area of the test specimen and by the modulus of elasticity of the material.

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[2] Instructor, Department of Engineering Mechanics, School of Engineering, The Pennsylvania State College, State College, Pa.

[3] R. K. Bernhard, "Automatic Speed Control for Tension and Compression Testing Machines," ASTM BULLETIN, No. 106, October, 1940, p. 31.

[4] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

Valuable suggestions have been given by Jones and Moore to facilitate the application of strain-rate specifications under various conditions. These show that no serious complication is introduced by the proposed change in specifications and testing procedure.

MR. L. B. TUCKERMAN.[5]—I have only one minor criticism. In commenting on mechanical testing machines of the screw-driven type, Jones and Moore state: "The speed of head is fixed by the train of gears used, . . but not very closely controlled." Three years ago Stang and Sweetman[6] of our laboratory described a simple electrical circuit, for use with direct-current motors, which gives as close control on screw-driven machines as is available on the best of hydraulic machines. We were much dissatisfied with the coarseness of the speed control of our screw-driven machines and adopted this resistance method. A few dollars spent for a reliable resistance control made it possible to have as close and as smooth continuously variable speed control upon our screw-driven machines as upon any of our modern hydraulic machines. We could not use our old screw-driven machines for many purposes if we did not have that control. We have it on every screw-driven machine in our Engineering Mechanics Laboratory. I think that the lack of close speed control on screw-driven machines has been a considerable factor in the present trend toward hydraulically driven machines. Modern hydraulically driven machines with direct-reading dials are of course more convenient in other respects than the older screw-driven, beam-and-lever type machine. They are amply accurate and constant for the great majority of tests on materials and structures.

However, no hydraulic machine that I know of today has the constancy and accuracy of a well-made, well-kept and properly used screw-driven beam-and-lever machine. With close and smooth continuously variable speed control readily available it would seem that they might well be more widely used.

MESSRS. P. G. JONES[7] AND H. F. MOORE[7] (*authors' closure*).—I am very glad to see that Mr. Fry has clearly emphasized the fact, brought out by his tests as well as by the tests of Withey[8] and by Jones and Moore, that the speed of the testing machine head cannot be used directly as a criterion of rate of strain. His development of machine modulus is useful for measuring rate of strain below the yield point, that part of the stress-strain diagram for which the modulus of elasticity is approximately constant. It is not useful beyond the yield region, and for that it is better to measure rate of strain directly. The machine modulus is, strictly speaking, a modulus of a particular setup of machine, grips and specimen. It is modified by variations in grips and in size and material of specimen as well as by the characteristics of the machine itself.

The writer cannot agree with the statement:

> "The Yield Strength may serve a purpose similar to that of the Yield

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[5] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

[6] A. H. Stang and L. R. Sweetman, "Speed Control for Screw-Power Testing Machines Driven by Direct-Current Motors," ASTM BULLETIN, No. 87, August, 1937, p. 15.

[7] Instructor in Theoretical and Applied Mechanics, and Research Professor of Engineering Materials, respectively, University of Illinois, Urbana, Ill.

[8] Report of Research Committee on Yield Point of Structural Steel, *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 136 (1931).

> Point, but the two are entirely distinct. Confusion would have been avoided if the sponsor of Yield Strength had recognized that no real yield is involved and had not infringed so closely on the old established name. Our concern here is with the Yield Point, a physical characteristic of the sharp-kneed materials, and not with the arbitrary and artificial Yield Strength."

Mr. Fry seems to use the term "yield" as restricted to deformation occurring with no increase of stress. Strictly speaking there is "yield" even under *elastic* deformation. However, as commonly used "yield" refers to any inelastic deformation regardless of the suddenness of its occurrence or of the rate of change of stress accompanying it. In determining the yield strength by the offset method or other methods, certainly yielding is involved. It is true that an arbitrary amount of yielding or set is specified as a criterion of structural damage. Since it is clear that the yield point obtained by drop of beam (the "upper" yield point) or from a stress-strain diagram is markedly affected by speed of testing, it seems quite reasonable to say that the yield point is also an arbitrary characteristic of the sharp-kneed materials, since it depends not merely on the material but on the speed of testing and on relative stiffness of testing machine and specimens. Hence, the ordinary "drop-of-beam" yield point can hardly be considered a property of a material any more independent of arbitrary factors than is the yield strength. Mr. Fry has not mentioned the difference between upper and lower yield points. The lower yield point, the minimum value of the stress after the drop of the beam, is less affected by rate of speed of testing machine than is the upper yield point. For materials with a sharp-kneed stress-strain diagram this lower yield point is usually approximately equal to the yield strength as determined by the "offset" method. It can be obtained readily with self-indicating machines but not readily with beam-balance machines without an autographic attachment.

Mr. Fry speaks of a "true" yield point being difficult to obtain. It is, of course, quite true that the determination of the yield point depends upon the uniform distribution of stress over the test specimen. However, there is some yielding, that is, there is some plastic action, well below any true yield point or yield strength in any actual material.

Yielding in a material has been shown by the metallographic microscope to begin in various crystals, and any measure of elastic strength depends upon the aggregate of such intra-crystalline yielding or "slip" becoming great enough to produce a definite effect, whether by a sudden yield point or the more gradual yield of the materials without sharp-kneed stress-strain diagrams. Again the speaker wishes to state his belief that yield strength and yield point are very definitely related in that both are measures of structural damage by inelastic yielding of a material. Both have distinctly arbitrary values, but both may be useful for determining the limit of a practical elastic strength of a material, and both involve a very definite yielding of the material.

It is especially gratifying that these two papers indicate that the effect of speed of testing seems to be about the same as shown by the test results reported in both. That means that it may not be necessary in future studies of effect of speed of testing on the properties of any material to use slow and expensive methods to insure freedom from "machine effects."

Thanks are due to Mr. Tuckerman for his note on the speed control of screw-power testing machines. Probably special mechanical or electrical speed controls could be used on a-c. circuits.

The results from the Dow Chemical Co.'s laboratories give further evidence of this fact. Further information concerning the apparatus developed at the Pennsylvania State College Laboratories is awaited with interest, and thanks are due Messrs. Schmidt and Carey for their discussions.

MR. LAWFORD H. FRY[9] (*Author's closure*).—Mr. McVetty's remarks are appreciated, as is too the counsel and assistance he gave the author in some of the experimental work. He notes the fact that the "test machine modulus" is intended for use only below the yield point. The author thought that this was clear in the paper, but Mr. Moore seems to have missed this point.

Mr. Moore seems to view the paper as a whole with disapproval. This is his privilege, but exception must be taken to several statements which seem to indicate lack of care in reading the paper.

Moore takes issue with the statement that Yield Point and Yield Strength are entirely distinct, and attempts to argue that the former is as arbitrary as the latter. This seems to ignore the definite objective characteristics of the two curves in Fig. 1. In the material producing curve B, something happened at the jog in the curve so that conditions before were not the same as the conditions after. In curve A there is no point at which there is a definite change in the before and after conditions. On this basis there is a distinct physical difference between the two materials represented by the two curves, and consequently between the Yield Point of Curve B and the Yield Strength of Curve A.

Moore objects to the statement that yield strength involves no real yielding and suggests that the author intends to restrict the term "yield" to deformation with no increase in stress. The author would have made his point clearer by writing, "no real yield occurs *at* the yield strength." The author does not restrict "yielding" to mean sudden deformation or to mean deformation without change in stress. The term is used in the sense of a giving way which marks a difference between the before and after conditions. This is the normal meaning, c.f. Webster: "Yield, v.i., To give way, . . . to cease opposition, . . . as 'the door yielded,' 'and yield to mercy while 'tis offered you.' " On this basis the smooth curve material yields continuously, with no definite yielding *at* the yield strength, while the sharp-kneed material yields slightly below the yield point and yields definitely *at* that point. These considerations seem to dispose of Moore's suggestion "that the yield point can hardly be considered . . . any more independent of arbitrary factors than is the yield strength." It is true that the value found for the yield point of a material may be varied somewhat by varying the type of specimen or the type of machine or the rate of pulling, but with uniform conditions of testing and uniform material, the sharp-kneed materials give a drop of beam or a jog in the stress-strain curve which is entirely objective. The yield strength in the smooth curve materials has no objective existence. It is merely the stress corresponding to a perfectly arbitrary offset on the curve.

The author feels strongly that the Yield Point marks a definite change in the structure of the material, while no such change takes place at the Yield Strength. For this reason the similarity in the names seems unfortunate. It is suggested that yield strength might well be renamed Offset Strength. The full designation would include the amount of offset contemplated, so that a specification would set a value for, say, a 0.2 per cent offset strength. The author believes that a clear-cut separation between the terms used for the sharp-kneed and for the smooth curve materials would represent an improvement in metallurgy, in engineering, and in etymology.

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[9] Railway Engineer, Edgewater Steel Co., Pittsburgh, Pa.

Moore says that the author speaks of a true yield point being difficult to obtain. His remarks, which appear to be critical, are hardly pertinent. The author's reference to the "true yield point" was to the effect that yield point values obtained by drop of beam in routine testing would normally be lower than the true value. The reason for this is that any stress concentration in the specimen due to curvature of the specimen, to nonaxial loading, or to action of the grips will lower the apparent value of the yield point. If specimens of uniform material are tested with perfectly uniform stress distribution, or if special precautions are taken to measure the stress at which yielding starts, the value found for the stress at which measurable plastic yielding begins may reasonably be called the true yield point. In routine testing some stress concentration in the specimen is inevitable. Yielding will start at the point of maximum stress concentration and will spread rapidly through the specimen, allowing the beam to drop. Obviously the average stress in the specimen corresponding to the load at which the beam drops is lower than the stress at the point of maximum concentration at which yielding started and which was the true yield point.

Moore's remarks do not really bear on this question. He says: "However, there is some yielding, that is, there is some plastic action, well below any true yield point or yield strength in any actual material." This statement does not bear on the phenomenon under discussion, and its accuracy depends entirely on the meaning given to the indefinite adjective "some." With sufficient delicacy of measurement there is "some" plastic deformation in the so-called straight line portion of the stress-strain curve, but this has little real pertinence in discussing drop of beam values. In the sharp-kneed materials the distance between the straight-line portion of the curve and the jog which marks the yield point is usually short, as in Fig. 1, so that the plastic deformation between the straight line and the yield point is usually small and unimportant. In the smooth curve materials, the plastic deformation below the yield strength is generally equal to the specified offset, so that it is measurably important. It is evident that Moore's statement takes on a definite meaning only when the difference between yield point and yield strength is noted. This again calls attention to the distinct differences between yield point and yield strength and emphasizes the desirability of renaming the latter. If we speak of "yield point" and "offset strength," the names will be distinctive and at the same time properly descriptive.

Since this was written, I have been very much pleased to find that Tuckerman in his discussion of the paper by C. S. Smith entitled "Proportional Limit Tests on Copper Alloys,"[10] expresses exactly the same views.

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[10] See p. 864.

ATMOSPHERIC DURABILITY OF STEELS CONTAINING NICKEL AND COPPER

By N. B. Pilling[1] and W. A. Wesley[1]

Synopsis

The results of six series of atmospheric corrosion tests are presented which evaluate the durability of sheet specimens of low alloy steels containing nickel and copper. Rural, urban, industrial and marine atmospheres are represented at the test sites. The commercial significance of the data is broadened by the multiplicity of compositions tested. The oldest series of exposures is now in its fifteenth year. Another series consists of large specimens simulating some of the conditions to which roofing sheets are exposed in service. The effects of composition variables are studied in one series and the corrosion-time relationship in another. The results of a practical railroad coal car test are also made available.

A general conclusion from this work is that the beneficial effects of nickel upon the weathering resistance of steel increase with increasing nickel content and are enhanced by the addition of optimum amounts of copper. The superiority of nickel copper steels over copper steels is particularly pronounced in a marine atmosphere to which the latter do not respond well. The effects of manganese, silicon and carbon contents do not appear to be important but the presence of a moderate amount of phosphorus seems to be beneficial in nickel copper steels.

Attention given to the mechanical properties of some of the newer types of high tensile strength constructional steels has somewhat overshadowed consideration of the adequacy of their corrosion resistance. This factor can be a most important one in the economy of weight-saving sheet steels. Unfortunately efforts to simulate weathering under accelerating conditions are not convincing or safe and it is still necessary to conduct field tests of long duration in evaluating the atmospheric durability of metals. Due to the fact that the corrosion resistance of nickel and nickel-copper steels has been receiving attention at this laboratory since 1925 it is already possible to draw conclusions with some degree of confidence regarding the usefulness of nickel and copper additions. Unlike the well-established effect of copper alone, which reaches its maximum at somewhat below ½ per cent copper content, the beneficial action of nickel increases continuously with rise in nickel content throughout the range of pearlitic steels and permits a level of corrosion resistance to be reached far beyond that made available by copper alone. On the other hand copper improves still further the rust resistance of nickel steels.

The literature is rich in data upon the durability of copper steels but the effect of nickel in the low-alloy range has been seldom studied experimentally. Howe[2] showed as early as 1901 that a 3 per cent nickel steel corrodes at a substantially lower rate in the atmosphere than unalloyed steels. The first preparation of nickel-copper steels seems to have been that of Clamer[3] who demonstrated the usefulness of additions of nickel and copper together in producing a high strength steel but included only highly alloyed austenitic steels in his atmospheric exposure tests. Burgess and Aston[4] found a marked decrease in the rate of atmospheric corrosion of electrolytic iron when alloyed with as little as 0.27 per cent nickel, an effect which was even more pronounced with higher nickel contents. A low alloy nickel-copper steel was included in Kalmus and Blake's tests[5] before 1916. The conclusion was drawn that the addition of small amounts of copper and nickel to ingot iron significantly decreased its rate of weathering.

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[1] Director, and Assistant Director, respectively, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

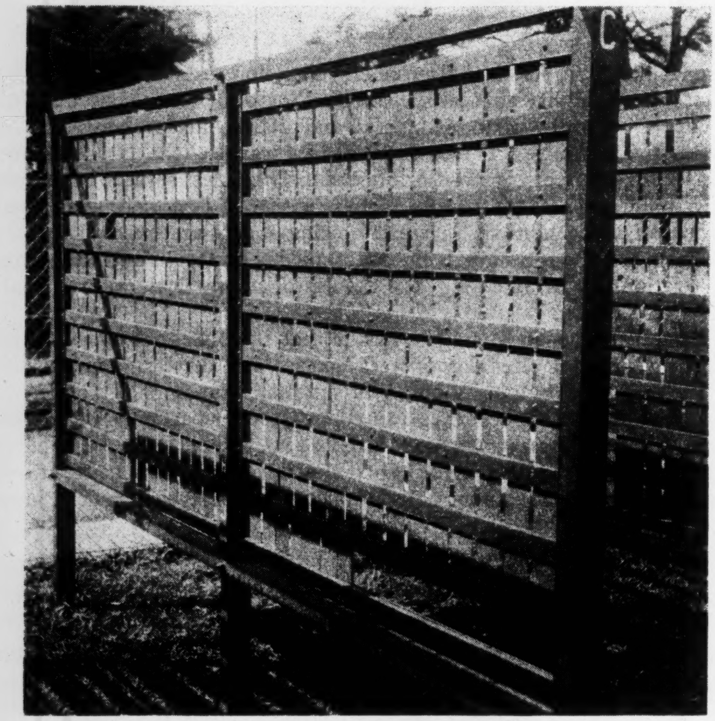

FIG. 1.—Vertical Atmospheric Exposure Rack.

A steel containing 3.75 per cent nickel was included in the world-wide marine tests of the Committee of the Institution of Civil Engineers. Atmospheric tests have been conducted at four seaports where losses in weight were measured at the end of 5, 10 and 15-yr. exposure.

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[2] H. M. Howe, "Effets relatifs de la corrosion sur le fer, l'acier doux et l'acier au nickel dans l'aire, l'eau de mer, et l'eau ordinaire," Congrés International des Méthodes d'Essai des Matériaux de Construction, Vol. 2, Part I, pp. 229 266 (1901).

[3] G. H. Clamer, "Cupro-Nickel Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. X, p. 267 (1910).

[4] C. F. Burgess and J. Aston, "Influence of Various Elements on the Corrodibility of Iron," *Transactions*, Am. Electrochemical Soc., Vol. 22, p. 241 (1912).

[5] H. T. Kalmus and K. B. Blake "Cobalt Alloys with Non-Corrosive Properties," *Report No. 411*, Mines Branch, Canada Department of Mines, pp. 7 and 35 (1916).

The committee[6] concluded from the 5 and 10-yr. results that "the addition of 3.75 per cent of nickel to 0.31 per cent carbon steel enhanced markedly its resistance to aerial and fresh-water corrosion." The 15-yr. results[7] further substantiate this conclusion.

A note[8] concerning some of the data to be described in the present report was contributed at the Symposium on High-Strength Constructional Metals held by the Society in 1936. At the same symposium Cone[9] stated that "During the past 10 yr., repeated confirmation has been secured of the beneficial effects of nickel in steels in contact with severely corroding atmospheres—either along the seacoast or in industrial centers and in certain other environments—in reducing the rate of attack and in changing the character of the corrosion products."

TABLE I.—WEATHERING TESTS BEGUN IN 1925.
Thickness of Specimens, 0.020 in.

Type	Chemical Composition, per cent					Life to First Perforation, yr.			
	Nickel	Copper	Manganese	Silicon	Carbon	Rural Atmosphere	Marine Atmosphere	Moderate Industrial Atmosphere	Heavy Industrial Atmosphere
Ingot iron	....	....	0.21	0.03	0.04	11	1.5	1.5	1.5
Copper steel (Commercial)	....	0.17	....	....	0.07	14.5	1.5	3.0	2.1
2 per cent nickel-copper steel	1.39	0.61	0.36	0.14	0.07	14.5	over 5	4	3.2
4 per cent nickel-copper steel	2.76	1.18	0.22	0.08	0.11	over 14.5	over 5	6	10
4 per cent nickel-copper steel	2.8	1.2	....	....	0.1	over 14.5	over 5	10	14.5
6 per cent nickel-copper steel	4.2	1.8	0.38	0.13	0.08	over 14.5	5	8	over 14.5
8 per cent nickel-copper steel	5.42	2.36	0.40	0.14	0.08	over 14.5	over 5	over 14	over 14.5
10 per cent nickel-copper steel	6.95	3.03	0.36	0.14	0.18	over 14.5	over 5	8[a]	over 14.5
10 per cent copper-nickel steel	3.0	6.71	0.40	0.18	0.05	over 14.5	3.5	5.5	12

[a] Failed by cracking.

Weathering Tests Begun in 1925:

Several experimental nickel-copper steels were included in an exploratory program started in 1924. The steels in question were made in a small high-frequency furnace from ingot iron and monel plus minor finishing elements. The ingots were forged to slabs and cold rolled to 0.020-in. strips which were finally annealed and ground bright.

One of the tests of corrosion resistance planned was exposure to various natural atmospheres. Four test sites were selected to represent atmospheres of different types. One was on a West Virginia farm some miles away from industrial activity. Another was on the beach in an isolated section of the New Jersey shore between Atlantic City and Beach Haven. A third rack was erected in the yard of this laboratory at Bayonne, not far from the center of oil-refining activity. The fourth test site was in a very smoky spot in Pittsburgh, Pa. adjacent to one of the railroad yards. On all racks the specimens were mounted in a vertical position facing south.

The test racks are similar to that illustrated in Fig. 1 and are designed to hold 3 by 5-in. sheet specimens. These are held at top and bottom in grooves in wooden cross members and are prevented from creeping and rattling in wind by asbestos cord used to caulk them tightly in the lower grooves which are about $\frac{1}{4}$ in. deep.

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[6] S. M. Dixon and H. J. Grose, "Deterioration of Structures of Timber, Metal and Concrete Exposed to Sea Water," Fifteenth Report of the Committee of the Institution of Civil Engineers, p. 49 (1935).

[7] J. N. Friend, "Losses in Weight and Pitting of the Committee's Iron and Steel Specimens After 15 Years Exposure," Seventeenth Report of the Committee of the Institution of Civil Engineers, pp. 12-26 (1938).

[8] Discussion by N. B. Pilling of paper by E. F. Cone, "Carbon and Low-Alloy Steels," Symposium on High-Strength Constructional Metals, p. 29, Pittsburgh Regional Meeting, Am. Soc. Testing Mats. (1936). (Symposium available as a separate publication.)

[9] E. F. Cone, "Carbon and Low-Alloy Steels," Symposium on High-Strength Constructional Metals, p. 1, Pittsburgh Regional Meeting, Am. Soc. Testing Mats. (1936). (Symposium available as a separate publication.)

Inspections after the second year have been made at intervals of approximately 1 yr. Upon these occasions records of failures and notes upon the general appearance of the rust coatings are made. Perforation of sheet by corrosion at a distance $\frac{1}{4}$ in. or more from the edges is considered the criterion of failure by corrosion. The life of the materials tested as the records stand to date is given in Table I. Not long after the fifth year the marine atmosphere exposure was terminated by a storm which washed the test rack out to sea.

Results.—The nickel-copper steels consistently demonstrated superiority over the copper steel in the sulfurous atmospheres and showed a high degree of resistance in the marine atmosphere under conditions to which the copper steel did not respond well.

Simulated Roofing Tests:

A more ambitious program was started in 1933 in which large samples were ex-

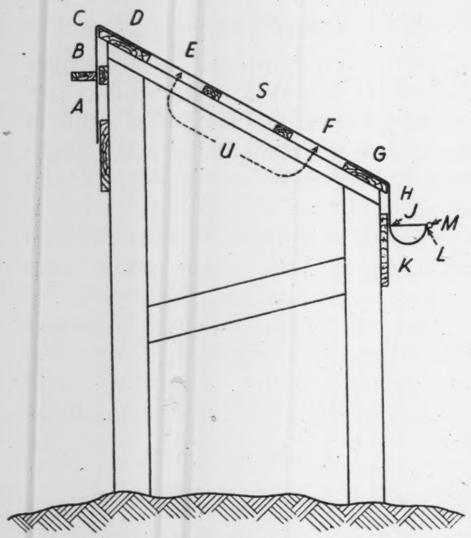

FIG. 2.—End View of Simulated Roofing Test Specimen.

(*A*) Vertical surface, partly sheltered from direct impingement of rain.
(*B*) Vertical surface, boldly exposed to rain.
(*C*) Sharp bend in sheet.
(*D*) Area having lower surface in contact with wood.
(*E*) and (*F*) Plane inclined surfaces.
(*G*) Area having lower surface in contact with wood.
(*H*) Vertical surface, boldly exposed.
(*J*) Area of water-line attact when trough is full of rain water.
(*K*) Area completely submerged when trough is full of rain water.
(*L*) Area of water-line attack.
(*M*) Trough bead.
(*S*) Lock seam, one-half soldered, one-half not.
(*U*) Fully sheltered lower surfaces.

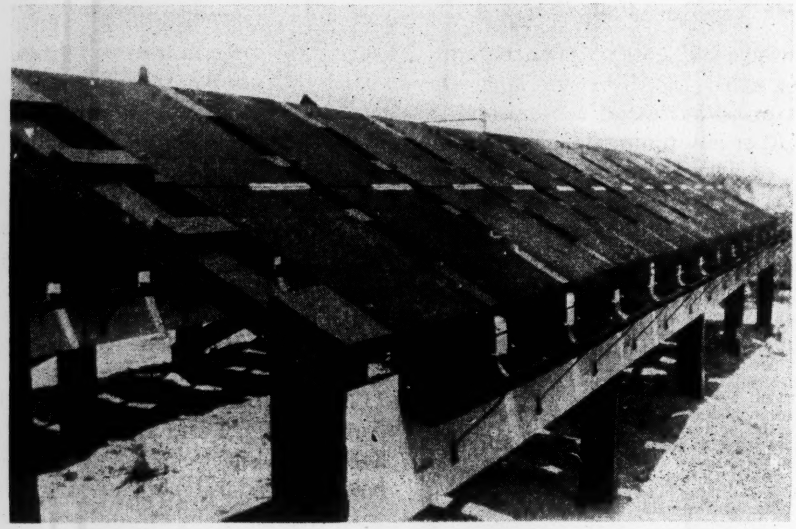

FIG. 3.—View of Simulated Roofing-Sheet Specimens, Fire Island, N. Y.

posed, fabricated to simulate some of the conditions to which roofing sheets are exposed in practice. The design of the specimens is evident in Figs. 2 and 3. It was expected to yield information on the relative corrosion resistances of various steels not only when exposed as plane inclined surfaces but also at areas sheltered from the rinsing action of rain, at seams, at sharp bends and in what might be called an alternate immersion exposure provided by an undrained rain trough. Each specimen was fabricated by a tinsmith from two 18 by 36 by 0.031-in. sheets. Thus the principal surface consists of an area 18 in. wide by 36 in. long supported at an angle of 30 deg. from the horizontal. This area slopes toward the front of the corrosion rack and terminates at the bottom in a section fabricated into a rain trough made watertight by soldered ends. Until corrosion results in leaks these troughs retain water for some time after rains.

Four test sites were selected to provide atmospheres of different types. One was in a pasture on a farm in central New Jersey, 7 miles from the nearest large town. Another was on Fire Island, N. Y. at a point about 200 yd. from the ocean beach. A third was in the yard of the laboratory in the moderately industrial atmosphere of Bayonne. The fourth was on the roof of a passenger train shed in Jersey City where the atmosphere is highly sulfurous from locomotive coal smoke. New York Harbor is a few hundred feet away, adding smoke from ships and tug boats and at times adding a slight salinity.

All the test racks face south.

TABLE II.—ANALYSIS OF MATERIALS IN SIMULATED ROOFING TESTS.

Type of Steel		Analysis, per cent						
		Nickel	Copper	Manganese	Silicon	Carbon	Phosphorus	Sulfur
Bessemer steel	Commercial	....	0.008	0.40	0.005	0.095	0.111	0.059
Ingot iron		....	0.06	0.02	0.001	0.015	0.008	0.026
Open-hearth copper steel		....	0.200	0.39	0.005	0.017	0.006	0.018
Rephosphorized open-hearth copper steel		....	0.252	0.49	0.005	0.065	0.052	0.022
Bessemer copper steel		....	0.212	0.40	0.018	0.100	0.112	0.059
Copper-molybdenum iron[a]		....	0.46	0.12	0.002	0.009	0.015	0.026
2 % nickel steel	Experimental	1.96	0.03	0.14	0.14	0.06	0.013	0.028
1 % nickel, 1 % copper		1.05	0.87	0.17	0.13	0.05	0.014	0.031
1.5% nickel, 1 % copper		1.58	0.86	0.32	0.20	0.05	0.043	0.068
2 % nickel, 1 % copper		1.77	0.93	0.13	0.13	0.05	0.012	0.030
1 % nickel, 2 % copper		1.24	2.23	0.39	0.63	0.08	0.017	0.033
2 % nickel, 2 % copper		2.16	1.97	0.28	0.52	0.06	0.018	0.005
3 % nickel, 1 % copper		3.12	1.14	0.20	0.21	0.08	0.015	0.009
3 % nickel, 1.3% copper		2.82	1.30	0.14	0.41	0.06	0.015	0.027
3 % nickel, 2 % copper		2.78	1.83	0.27	0.20	0.06	0.013	0.008
4.4% nickel, 2 % copper		4.40	2.05	0.24	0.20	0.11	0.019	0.008
5.5% nickel, 1.3% copper		5.53	1.31	0.17	0.15	0.08	0.012	0.007
6.4% nickel, 1 % copper		6.35	1.06	0.16	0.31	0 08		
6.4% nickel, 2 % copper		6.40	2.12	0.13	0.12	0.11	0.014	0.011

[a] Contains molybdenum, 0.09 per cent.

The analysis of the steels used in these exposures are given in Table II. The commercial bessemer and copper steels were secured from routine heats. The experimental nickel-copper steel ingots were made from 450-lb. or 1100-lb. arc furnace melts. The ingots were forged, hot-rolled to 0.40-in. sheets, annealed, pickled, and cold-rolled to 0.033 in. After a box anneal the sheets were cut to specimen size and were ready for fabrication after a pickling and scrubbing operation.

Results to Date.—The dates on which inspections were made and the record of failures due to corrosion are shown in

TABLE III.—SIMULATED ROOFING TESTS. TIME OF INITIAL FAILURE AT DIFFERENT AREAS OF EACH SPECIMEN.

Blank spaces mean no failure at time of last inspection. Letters refer to areas designated in Fig. 2. Results show life in months. Average initial thickness of sheets was 0.033 in. See Table II for chemical analyses.

Type of Steel	Rural Atmosphere							Marine Atmosphere							Industrial Atmosphere							Sulfurous Atmosphere							Average Lives of Gutters (Areas *JKLM*) Exclusive of Rural Atmosphere
	Inspected at 15, 25, 38, 62, 74, 83 and 94 months							Inspected at 4, 15, 25, 30, 37, 45, 61 and 73 months							Inspected at 4, 7, 12, 16, 18, 23, 25, 26, 29, 38, 45, 62, 73, 76, 79, and 94 months							Inspected at 4, 10, 12, 22, 34, 42, 58, 73, 84, and 90 months							
	A	*B*	*C* to *G*	*J*	*K*	*L*	*M*	*A*	*B*	*C* to *G*	*J*	*K*	*L*	*M*	*A*	*B*	*C* to *G*	*J*	*K*	*L*	*M*	*A*	*B*	*C* to *G*	*J*	*K*	*L*	*M*	
Bessemer 0.01% Cu, 0.11% P	74		62	25	15	25	25	15[a]	15[a]	15[a]	15[a]	15[a]	15[a]	15[a]	18	18	25	12	12	12	12	12	12	12	12	12	12	12	13
Ingot iron 0.06% Cu, 0.01 P				74	62	74		25	61		25	15	15	25				25	29	29	25	42	58	34	12	22	22	22	22
Open-hearth copper 0.20% Cu, 0.01% P				74	25	74		37	61		61	25	25	15				25	25	25	25	58	58	58	12	22	22	22	25
Rephosphorized open-hearth copper 0.25% Cu, 0.05% P					62			25			61	15	15	25				26	29	26	25	58	73	84	34	34	22	22	27
Bessemer copper 0.21% Cu, 0.11% P					62			25	61		61	25	25	25				38	38	38	26	73	73	84	12	34	22	22	31
Copper-molybdenum iron 0.46% Cu, 0.01 P					62			25			61	15	15	25				38	29	29	27	58	73	84	34	22	22	22	28
2 % Ni, 0 % Cu					62						61	25	25	37				38	29	26	26	58			34	34	22	34	33
1 % Ni, 1 % Cu					62			61			61	37	30	37				38	29	26	26	73			22	34	22	34	33
1.5% Ni, 1 % Cu				74	62	74					30	37	30	37				38	29	29	26				34	34	22	22	31
2 % Ni, 1 % Cu				74	62	74					61	25	37	45				38	29	29	27				34	34	22	22	34
1 % Ni, 2 % Cu					62						30	37	37	37				26	29	26	26				22	34	34	22	30
2 % Ni, 2 % Cu					62						30	37	37	37				38	29	26	29				34	34	22	22	31
3 % Ni, 1 % Cu					62						61	37	37	45				29	29	38	26				34	34	34	22	36
3 % Ni, 1.3% Cu					62						61	37	45	61				38	29	38	38				34	34	34	22	39
3 % Ni, 2 % Cu					62						61	37	37	45				29	38	29	38				34	34	34	34	38
4.4% Ni, 2 % Cu					62						61	37	37	61				29	38	38	38				42	34	34	34	40
5.5% Ni, 1.3% Cu					62						61	37	61	61				29	29	26	26				34	34	34	22	38
6.4% Ni, 1 % Cu					62						61	45	45	61				38	45	45	45				34	42	42	42	45
6.4% Ni, 2 % Cu					62						61	45	45	61				29	38	38	38				73	34	34	22	42

[a] Condition indicates failure occurred in considerably less than 15 months.

Table III. As before, failure is defined as perforation of the sheet by corrosion at any point more than $\frac{1}{4}$ in. removed from the edges. Directing attention first to the trough areas *JKLM* which represent rain gutters, it will be noted that these had all completely failed at the end of 6 yr. except those in the rural atmosphere. The corrosive conditions are much more severe than simple weathering and constitute a sort of alternate immersion test. The data in the last column of Table III show that the copper steels were much superior to the Bessemer steel and that phosphorus additions were definitely beneficial in the presence of copper. In the nickel-copper steels an excess of nickel over copper content was required to develop a degree of resistance to this "wet and dry" corrosion much superior to that of the copper steels.

In the areas of the roofing specimens subjected to simple weathering, failures usually occur earlier in area *A*, where both surfaces of the steel are sheltered from rain, than in areas *B* to *G*, inclusive, where only the under surface is sheltered. The data confirm the beneficial effect of copper. The improvement effected by nickel additions is also evident but the magnitude of the superiority of the nickel-copper steels cannot yet be evaluated because none of the steels containing more than 2 per cent alloy has yet failed. The advantage is probably greatest in a marine atmosphere where the copper steels failed so early that the 2 per cent nickel steels and the nickel-copper steels containing more than 2 per cent alloy had already outlasted the copper steels 200 to 300 per cent at the end of 6-yr. weathering. Unfortunate'y, the record at the seacoast site at Fire Island, N. Y. cannot be completed because the testing station was destroyed by the sea during the hurricane of September, 1938.

TESTS OF A NICKEL-COPPER STEEL IN COAL CARS AND IN SOILS

By 1931 it had become clear that nickel in small percentages was beneficial to the resistance of sheet steel to atmospheric corrosion, and the data then available suggested that the rate of improvement was within the limits of economic interest. Consideration of the balance between cost and greater life, between increased mechanical properties and ease of working, lead to the selection of 2 per cent nickel, 1 per cent copper as a type of composition for trial in two unusual opportunities which became available at that time. These were a new campaign of soil corrosion burials by the National Bureau of Standards, and the repair of some seventy hopper coal cars on one of the railroads.

The railroad coal cars were of 50-ton capacity and normally in use transporting bituminous coal. It was proposed to repair the droppers and bottoms using the nickel-copper steel in one half of each car, and ordinary open-hearth or copper-bearing steel in the opposite end for comparison. These tests were under the direction of H. J. French and A. L. Roberts of The International Nickel Co. Several experimental open-hearth heats of nickel-copper steel were made which were hot-rolled into $\frac{1}{4}$-in. plate; the carbon and copper-bearing steel plates were from commercial stocks.

A typical analysis of the steel going into this experiment was

Carbon, per cent	0.15
Manganese, per cent	0.52
Phosphorus, per cent	0.010
Sulfur, per cent	0.024
Silicon, per cent	0.21
Nickel, per cent	2.06
Copper, per cent	1.13

The identification of carbon and copper-bearing plates was established by analysis after installation in the cars.

The cars went into service in 1932 and ten of them were given approximately yearly inspections for rate of thinning of the sheets. This measurement was obtained by drilling holes, 1.19 in. in diameter near the bottom of the inclined sheets, cleaning off the scale and micrometering. The hole was then closed with a rivet.

The result of four successive examinations is given:

CUMULATIVE THINNING OF COAL CAR HOPPER PLATES.

Average, per cent			Number of Plates	Average Loss in Thickness of Plates, in.			
Copper	Nickel	Phosphorus		1.5 yr.	2.9 yr.	4.2 yr.	5.8 yr.
0.047	<0.03	0.018	14	0.015	0.030	0.044	0.056
0.25	<0.03	0.018	6	0.015	0.024	0.034	0.043
1.04	2.01	0.011	20	0.005	0.011	0.019	0.026

Six years of a service which included the shock and abrasion of repeated loadings and unloadings, weathering and contact with the acid leachings from soft coal, resulted in rates of thinning which were nearly proportionate to the lapse of time. The nickel-copper steel plates suffered on the average only 50 per cent of the deterioration shown by copper-bearing steel, which in turn was more resistant than the carbon steel.

TABLE IV.—ANALYSIS OF BUREAU OF STANDARDS SOIL CORROSION TESTS: 5.5-YR. DURATION.

Steel	Composition, per cent									Average Weight Loss by Corrosion, oz. per sq. ft.				Average Depth of Deepest Pits, in.[a]		
	Carbon	Manganese	Silicon	Phosphorus	Sulfur	Copper	Nickel	Chromium	Molybdenum	5 Acid Soils	5 Moderately Acid Soils	4 Alkaline Soils	Cinders	5 Acid Soils	5 Moderately Acid Soils	4 Alkaline Soils
No. 1	0.15	0.49	....	0.013	0.030	....	....	....	....	1.25	1.32	2.83	6.59	0.050	0.077	0.096
No. 2	0.04	0.32	0.05	0.016	0.027	0.52	....	....	0.15	0.93	1.07	2.75	4.76	0.045	0.082	0.098
No. 3	0.13	0.46	....	0.012	0.025	....	....	5.05	....	1.02	1.07	2.84	5.20	0.068	0.083	0.096
No. 4	0.14	0.21	0.19	....	0.012	1.08	2.47	....	....	0.81	0.72	2.03	4.47	0.033	0.037	0.061

[a] All 4 steels were perforated by pitting in cinders.

Also in 1931, the National Bureau of Standards was preparing for a new series of corrosion tests on tubular ferrous materials buried in a variety of natural soils; nickel-copper steel in the 2 per cent nickel, 1 per cent copper proportion was submitted for inclusion. This material was supplied from experimental basic arc-furnace heats which were forged, hot-pierced and cold drawn to $1\frac{1}{2}$-in. iron pipe size tubing having a wall thickness of 0.25 in. The protective end caps to the tubular specimen were made from sand castings from a separate heat of the same type of steel.

Several reports have been published by the Bureau of Standards on the results from this program, the latest of which[10] deals with exhumations after 5.5-yr. burial. The data in Table IV have been taken from that publication. The selection of steels for comparison and the averaging of data are our own; the grouping of soils is that developed on p. 526 of the paper cited. Those who may be interested in this are urged to study the complete data given in the reference cited.

These data clearly suggest, at the present stage of test, an advantage for the nickel-copper steel in resistance to general corrosion and pitting under several conditions of soil contact; it may be remarked that similar trends have not been noted by us in submerged corrosion tests in water.

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[10] K. H. Logan, "Soil Corrosion Studies, 1937," *Journal of Research*, Nat. Bureau Standards, Vol. 23, p. 515 (1939).

EFFECT OF VARIABLES IN COMPOSITION (1933 SERIES)

A quickening interest in nickel copper steels stimulated need for more information on the effects of composition factors on resistance to weathering. An extensive series of experimental nickel-copper steels was prepared in 0.015-in. gage and exposed as simple rectangular panels in the Bayonne industrial atmosphere. This thinner gage was selected in order to shorten the time to failure. Confining the exposure test to a simple specimen shape and to one atmosphere permitted concentration of effort upon a large number of combinations of nickel, copper, and minor element contents.

TABLE V.—ANALYSIS OF REPRESENTATIVE SPECIMENS FOR 1933 SERIES.

A. Complete Analysis of Several Typical Heats

Nominal Composition, per cent						Actual Analysis, per cent						
Nickel	Copper	Manganese	Silicon	Carbon	Phosphorus	Nickel	Copper	Manganese	Silicon	Carbon	Phosphorus	Sulfur
0	0	0.3	0.1	0	0	0.05	0.08	0.28	0.06	0.03	0.006	0.030
2	1	0.3	0.1	0	0	2.04	0.95	0.27	0.06	0.15	0.006	0.026
2	1	2.0	0.1	0	0	1.96	1.03	1.85	0.06	0.17	0.012	0.031
2	1	0.3	0.1	0	0.12	2.01	1.01	0.29	0.16	0.17	0.12	0.033

B. Variation from Nominal Composition

Element	Number of Heats Analyzed	Composition, per cent		
		Nominal	Determined	
			Average	Standard Deviation[a]
Nickel	34	2	2.000	0.064
	27	1	1.038	0.044
	49	0	Less than 0.05	
Copper	23	2	2.063	0.049
	27	1	1.060	0.040
	22	0.5	0.556	0.022
	12	0	0.06	

[a] Manual on Presentation of Data, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 453 (1933).

Some 200 melts were prepared in a high-frequency induction furnace using an Armco iron base. They comprised 12 series ranging from 0 to 4 per cent nickel and 0 to 2 per cent copper; each of these as a base additionally ranged 0.3 to 2.0 per cent manganese 0.1 to 1.0 per cent silicon, 0.05 to 0.4 per cent carbon, and 0.01 to 0.12 per cent phosphorus. Each melt was killed with 0.02 per cent aluminum. Slices cut from each ingot were then cold-rolled with intermediate anneals and pickling treatments, as required, to 0.015-in. strip. The final heat treatment was for 3 hr. at 1550 F. followed by furnace cooling.

Procedure in preparing these steels was so standardized that it was not necessary to have the melts completely analyzed. Determinations of nickel and copper were made with almost all of the strips with the results shown in Table V. Complete analyses of four typical steels of this series are included.

To aid in the comparison of results of this experiment with those of the simulated roofing tests, strips were cut from each of the steels of Table II. These were cold-rolled to 0.015-in. gage, annealed at 1550 F. and pickled.

The corrosion test specimens were exposed as 3 by 4.9 by 0.015-in. panels,

TABLE VI.—EFFECTS OF COMPOSITION VARIABLES, 1933 SERIES.

				Life of 0.015-in. Strips at Bayonne, N. J., months											
Nickel, per cent......				0	0	0	0	1	1	2	2	2	4	4	4
Copper, per cent.....				0	0.5	1	2	0	0.5	0	1	2	0	1	2
Manganese, per cent	Silicon, per cent	Carbon, per cent	Phosphorus, per cent												
0.3	0.1	0	0	14	24	31	35	49	37	31	over 83	59	59	over 83	72
0.6	0.1	0	0	[a]	15	24	15	26	59	31	37	35	over 83	over 83	over 83
1.0	0.1	0	0	35	46	59	28	28	31	37	37	37	[a]	over 83	over 83
2.0	0.1	0	0	[a]	26	52	31	31	63	72	over 83	[a]	[a]	[a]	[a]
0.3	0.2	0	0	19	26	31	31	28	37	37	44	40	over 83	over 83	over 83
0.3	0.5	0	0	24	[a]	28	31	44	31	[a]	72	59	over 83	over 83	over 83
0.3	1.0	0	0	28	28	31	[a]	46	35	37	[a]	59	over 83	over 83	over 83
0.3	0.1	0.2	0	[a]	over 83	31	31	28	31	40	63	44	72	over 83	over 83
0.3	0.1	0.3	0	26	63	28	37	59	28	31	72	37	over 83	over 83	over 83
0.3	0.1	0.4	0	[a]	26	31	52	31	over 83	25	79	44	79	over 83	over 83
0.3	0.1	0	0.03	[a]	[a]	59	52	37	37	44	59	59	over 83	over 83	over 83
0.3	0.1	0	0.06	28	[a]	46	72	over 83	over 83	over 83	46	[a]	over 83	over 83	over 83
0.3	0.1	0	0.09	28	[a]	59	44	over 83	37	40	over 83	over 83	over 83	over 83	over 83
0.3	0.1	0	0.12	44	[a]	40	28	37	44	40	over 83	44	over 83	over 83	over 83

[a] This composition not represented in test.

TABLE VII.—OTHER RESULTS FROM 1933 SERIES.

Specimens of the steels from the Roofing Test of Table II were rolled to 0.015-in. gage and were exposed at same time as experimental steels of Table VI. Atmospheric exposure at Bayonne, N. J.

Type of Steel	Life, months
Bessemer 0.01% Cu, 0.11% P.............	12
Ingot iron 0.06% Cu, 0.01% P............	24
Open-hearth copper 0.20% Cu, 0.01% P...	35
Rephosphorized open-hearth copper 0.25% Cu, 0.05% P..................	42
Bessemer copper 0.21% Cu, 0.11% P......	35
Copper-molybdenum iron 0.46% Cu, 0.01% P..................	31
2 % Ni, 0 % Cu.........................	59
1 % Ni, 1 % Cu.........................	46
1.5% Ni, 1 % Cu.........................	59
2 % Ni, 1 % Cu.........................	59
1 % Ni, 2 % Cu.........................	37
2 % Ni, 2 % Cu.........................	72
3 % Ni, 1 % Cu.........................	More than 83
3 % Ni, 1.3% Cu.........................	More than 83
3 % Ni, 2 % Cu.........................	More than 83
4.4% Ni, 2 % Cu.........................	More than 83
5.5% Ni, 1.3% Cu.........................	More than 83
6.4% Ni, 1 % Cu.........................	More than 83
6.4% Ni, 2 % Cu.........................	More than 83

in an annealed condition and with a clean, pickled surface. They were held in a vertical position in wooden racks similar to those described earlier for the 1925 tests. In the early stages inspections were made every few months, later about twice yearly. Perforation of a specimen by corrosion at a point removed more than $\frac{1}{4}$ in. from the edges was considered failure and so recorded.

Effects of Manganese, Silicon and Phosphorus:

The record for most of the steels can be conveniently and concisely presented in the systematic arrangement of Table VI. Data for steels which cannot be readily fitted into this arrangement are assembled in Table VII. Considering the elements manganese, silicon and carbon first, one is tempted to conclude that higher manganese contents give erratically higher life, while higher silicon and higher carbon have no effects. Analysis of the data, however, indicates that there is no statistical basis for concluding that any of these elements exerted a significant effect. On the other hand the large proportion of specimens which have not yet failed after 83 months exposure in the group of steels containing 0.03 to 0.12 per cent phosphorus is strong indication that this element exerts a beneficial effect when added to nickel and copper steels.

Effects of Nickel and Copper:

Statistical analysis is not needed to establish that an improvement is conferred by nickel and copper additions. With these there is a sweeping trend clearly over-riding the variations of other elements of composition. This is made clear by Fig. 4 which includes the results with not only the steels of Table VI but those of Table VII. Exceptions to the general trend occur when the ratio of copper to nickel contents is too high. Thus the 1 per cent nickel, 2 per cent copper alloy of Table VII is inferior to the other nickel copper steels and the 2 per cent nickel, 2 per cent copper is inferior to the 3 per cent nickel, 1 per cent copper steel.

In attempting to express the results of these tests quantitatively it is evident that this cannot be done by calculating the average life for steels with each combination of nickel and copper content because many of the steels have not yet failed. Again, a simple average does not eliminate the errors due to time elapsed between the occurrence and the recording of a given failure. Passano and Hayes[11] suggest that data of this sort can best be evaluated by determination of the "most probable life" of a group of specimens, namely, the time of exposure at which 50 per cent of the sheets in the group have failed. The effect of frequency of inspection is minimized by plotting a "time *versus* percentage failures" curve on Hazen probability graph paper. If the data show the usual probability

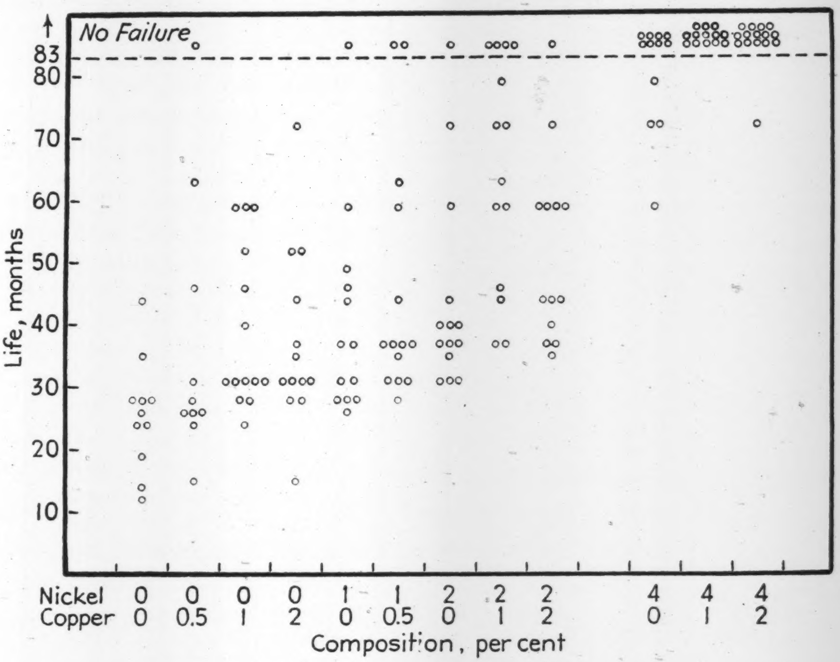

FIG. 4.—Effect of Nickel and Copper, 1933 Series.

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[11] R. F. Passano and Anson Hayes, "A Method of Treating Data on the Lives of Ferrous Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 220 (1929).

distribution a straight line is obtained on this paper. The point of intersection of the straight line with the 50 per cent mark corresponds to the maximum of the probability function (or where the largest number of samples failed) which is the most probable life.

Most of the groups of data of Fig. 4 when plotted upon Hazen probability paper diverged from a straight line, particularly at low and high values of percentage failures, but it was felt that the best line drawn through points near the 50 per cent mark yielded a value for most probable life which does indeed have quantitative significance.

TABLE VIII.—1933 SERIES: ESTIMATE OF MOST PROBABLE LIFE.

The "most probable life" is defined by Passano and Hayes [11] as the time at which 50 per cent of the sheets of a given class have failed.

Hazen probability graph paper was used in estimating this time.

Composition, per cent		Number of Samples Exposed	Most Probable Life, months
Nickel	Copper		
0	0	11	24
0	½	9	26
0	1	14	32
0	2	13	32
1	0	13	32
1	½	14	37
2	0	14	37
2	1	14	62
2	2	13	43
4	0	12	More than 85
4	1	13	More than 85
4	2	13	More than 85

The results of these determinations are presented in Table VIII. From them and from Table VII can be drawn the following conclusions:

(*a*) The beneficial effect of nickel added alone increases continuously with increase in nickel content.

(*b*) Addition of copper to nickel steels has a beneficial effect upon durability but a maximum effect is achieved at a copper content probably somewhat less than the nickel content.

(*c*) Addition of nickel to copper steels increases their durability. Greenidge and Lorig[12] concluded that 0.4 per cent nickel added to copper steels is not very effective but the present results show that a nickel addition of 1.0 per cent accomplishes marked improvement.

Corrosion-Time Relationship

A convenient method for determination of change in rate of corrosion with time is the electrical resistance method which has been employed successfully by various workers studying the corrosion of non-ferrous metals, for example, by Burns and Campbell.[13] It did not seem desirable to follow the customary procedure of employing wires as test specimens because wires could not be expected to duplicate the weathering behavior of sheet steel which forms so much coarser a coating of rust. Instead specimens were prepared from some of the 0.015-in. steel sheets in the form of strips 0.50 in. wide. Each end of the strip was firmly attached to a heavy copper lug so that the length whose resistance was to be measured was 10.5 in. and would remain constant at this value. The ends were lacquered and painted to prevent galvanic effects. The initial resistance (R_o) of each specimen was carefully measured at 100 F. and the test specimens were exposed on an unsheltered rack at Bayonne in such a position that the long dimension was in a horizontal direction, but the surfaces of the strip were vertical. Each time that a measure of the amount of corrosion was desired, the specimens were brought indoors, allowed to dry at room temperatures and then were subjected to another measurement of resistance (R_t) at 100 F. They were then restored to the exposure rack without

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[12] C. T. Greenidge and C. H. Lorig, "Copper Steels," *Iron Age*, Vol. 145, No. 7, p. 21 (1940).

[13] R. M. Burns and W. E. Campbell, "Electrical Resistance Method of Measuring Corrosion of Lead by Acid Vapors," *Transactions*, Electrochemical Soc., Vol. 55, p. 271 (1929).

TABLE IX.—CORROSION-TIME RELATIONSHIP

Decrease in thickness of 0.015-in. steel strips measured by change in electrical resistance during exposure to the Bayonne atmosphere. Decrease in thickness = $\frac{R_t - R_o}{R_t} \times 0.015$ in.

Material	Analysis, per cent							Decrease in Thickness, in.						Estimated Time for Decrease Equal to 0.0075 in., months
	Nickel	Copper	Manganese	Silicon	Carbon	Phosphorus	Sulfur	End of 4.6 months	End of 7.5 months	End of 12.5 months	End of 24.7 months	End of 33.0 months	End of 76.4 months	
Ingot iron	.....	0.06	0.02	0.001	0.015	0.008	0.026	0.00281	0.00360	0.00530	0.00765	0.00882	[b]	24
Copper-molybdenum iron[a]	.....	0.46	0.12	0.002	0.009	0.015	0.026	0.00309	0.00389	0.00488	0.00630	0.00695	0.00926	41
Experimental steel	1.96	0.03	0.14	0.14	0.06	0.013	0.028	0.00234	0.00290	0.00336	0.00501	0.00582	0.00783	66
	1.77	0.93	0.13	0.13	0.05	0.012	0.030	0.00225	0.00275	0.00363	0.00438	0.00513	0.00689	98
	3.12	1.14	0.20	0.21	0.08	0.015	0.009	0.00269	0.00290	0.00348	0.00431	0.00483	0.00633	130

[a] Contains molybdenum, 0.09 per cent.
[b] Strip corroded through at several points and broken at one.

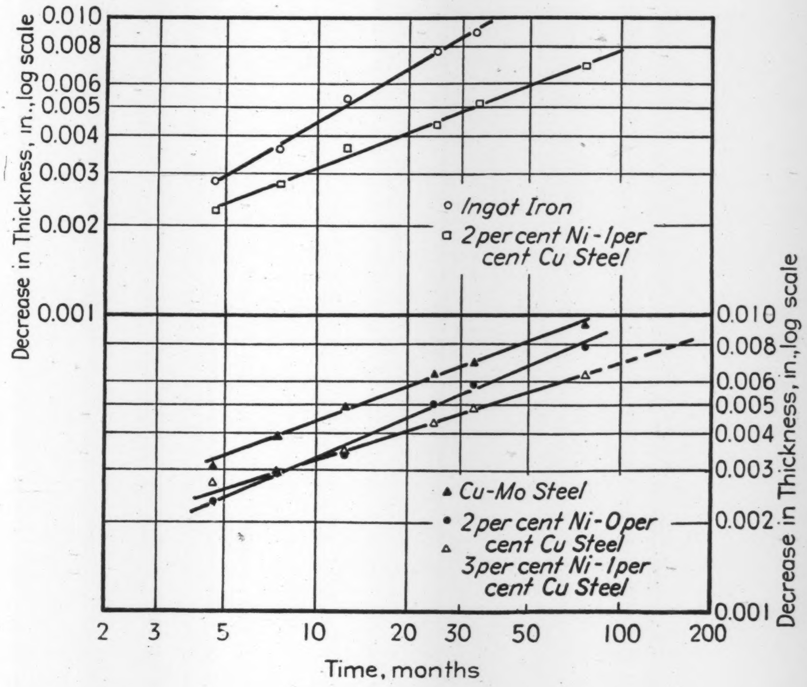

FIG. 5.—Corrosion-Time Relationship.

disturbing the rust coating which had formed to date.

Decrease in thickness of the test strips was computed from the change in resistance by using the simple formula:

$$\frac{R_t - R_o}{R_t} \, 0.015 \text{ in.}$$

The results of this experiment thus far are presented in Table IX and Fig. 5.

Discussion of Corrosion-Time Relationship:

The relationship between the logarithm of the amount of corrosion and the logarithm of time of exposure in Fig. 5 is a straight line, as were Passano's[14] curves for plain and copper steels. An important conclusion to be drawn from the present results is that the superiority demonstrated by the alloyed steels becomes progressively greater with increase in duration of the exposure, hence with increase in gage of the metal being tested.

The more rapid decrease in rate of corrosion with time with the alloyed steels is related to the structure and protective value of the rust coatings. Steels with rapid rates of corrosion developed very coarse-textured rust coatings while the more resistant steels were readily recognized by the fineness and good adherence of their rust.

TABLE X.—ATMOSPHERIC AND SEMI-IMMERSION TESTS AT BAYONNE STARTED IN 1934.

In the semi-immersion or water-line tests clean sheet specimens 5 in. square were exposed outdoors half immersed in an open unsheltered trough. The water level was maintained by use of an inverted bottle of distilled water as a constant level device.

Steel	Chemical Analysis, per cent									Loss in Weight, mg. per sq. dm per day				
										Atmospheric		Semi-Immersion		
	Nickel	Copper	Manganese	Silicon	Carbon	Phosphorus	Sulfur	Molybdenum	Chromium	12 6 months	36.8 months	5.7 months	13.9 months	20 months
No. 1	...	0.06	0.02	0.001	0.02	0.008	0.026	...	...	20.7	13.9	18.9	23.4	18.8
No. 2	0.03	0.12	0.36	...	0.10	0.037	0.027	...	...	14.5	9.8	19.3	21.7	17.5
No. 3	...	0.29	0.30	<0.01	0.03	0.008	0.053	...	...	14.5	8.2	18.3	21.6	16.5
No. 4	...	0.60	...	...	0.028	0.011	...	0.094	...	13.8	7.1	21.8	17.9	15.3
No. 5	0.09	1.25	0.64	0.01	0.20	0.017	0.018	0.30	...	13.3	7.3	19.8	17.8	15.7
No. 6	...	...	0.52	0.15	0.21	0.017	0.024	0.22	...	16.9	9.5	27.4	24.2	21.9
No. 7	0.05	0.45	0.33	0.79	0.13	0.15	...	...	1.15	9.7	...	24.3	15.1	14.7
No. 8	0.48	0.50	0.46	<0.01	0.12	0.011	0.029	...	0.41	12.7	7.0	17.3	21.8	19.2
No. 9	1 37	...	0.70	0.91	0.19	0.015	0.025	...	...	13.5	7.5	26.6	16.9	19.2
No. 10	2.07	...	0.37	...	0.07	0.011	0.029	...	...	10.9	6.6	19.1	20.0	16.5
No. 11	1.58	0.86	0.32	0.20	0.05	0.045	0.069	...	...	10.2	5.9	...	15.6	13.6
No. 12	1.74	0.09	0.36	0.20	0.125	0.018	0.025	0.24	...	9.8	6.1	19.6	17.3	16.7
No. 13	5.03	...	0.49	0.30	0.13	0.018	0.019	...	...	7.1	4.2	16.0	13.5	13.1

ATMOSPHERIC AND SEMI-IMMERSION TESTS STARTED IN 1934

A group of 11 alloy structural steels was exposed to the Bayonne atmosphere in 1934. The specimens were 2.5 by 5 in. in size, cut from 22 gage, clean, pickled sheet. These were exposed in two ways: one set was mounted on a boldly exposed rack similar to that shown in Fig. 1; the second set was exposed outdoors in a vertical position and half immersed in water in an open unsheltered trough. The trough was kept full of water by rain and by use of an inverted bottle of distilled water as a constant level device. Each steel was exposed in triplicate and it was planned to remove one piece of each trio from test at the end of 1, 3 and 8 yr., respectively.

The first two periods of test have been completed for the atmospheric exposure and the semi-immersion test was terminated at the end of 20 months. The weight changes are given in Table X. Although the losses of the partially immersed specimens were about double those in the atmospheric tests the damage done to the former was much greater because there was localization of attack near the water level or at the edges of the specimens. The more highly alloyed steels were definitely superior to the others in the partial immersion test.

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[14] R. F. Passano, "The Harmony of Outdoor Weathering Tests," Symposium on the Outdoor Weathering of Metals and Metallic Coatings, p. 28, Washington Regional Meeting, Am. Soc. Testing Mats. (1934). (Symposium available as a separate publication.)

In the atmospheric tests the more highly alloyed steels were again superior to the others in resistance to corrosion. The third set of these steels is still exposed. The marked drop in corrosion rate with time in the first 3 yr. indicates that tests of this sort should be continued for many years if the true relative longevity of alloy steels is to be established.

THE EFFECT OF PROTECTIVE COATINGS ON THE CORROSION-FATIGUE STRENGTH OF STEEL

By Thomas J. Dolan[1] and Hugh H. Benninger[1]

Synopsis

To examine the relative efficiency of protective coatings in increasing the corrosion-fatigue strength of steels, laboratory tests were made of the following types of protective coatings:

1. A quenched and tempered S.A.E. 3140 steel with thin electroplated cadmium and zinc coatings and also a cyanided surface.
2. A silicon impregnated case on S.A.E. 1020 steel.
3. A nitrided case on S.A.E. 6120 steel.

All specimens were subjected to completely reversed cycles of flexural stress while in contact with fresh tap water. It was found that the corrosion-fatigue endurance limits for all coatings except the silicon impregnation were considerably below the endurance limits of the uncoated material in air. The specimens with the silicon impregnation coatings exhibited the same endurance limits in air and in water, but the value of the endurance limit was only about 22,500 psi.

The zinc-plated specimens had a somewhat higher corrosion-fatigue endurance limit than did the cadmium plated specimens but both platings gave endurance limits less than 45 per cent of the endurance limit of the uncoated material in air.

The chief effect of the electroplated coatings seemed to be a lengthening of the time (and number of cycles of stress) required to cause failures of the coated specimens in corrosion-fatigue.

The nitrided case was very efficient in resisting pitting due to corrosive attack and the corrosion-fatigue endurance limit of the nitrided specimens was the highest obtained (85,000 psi.) from any of the materials tested. Several tests of specimens coated with bakelite varnishes indicated that an increased corrosion-fatigue strength would be developed if a continuous impervious coating can be maintained.

A serious reduction of the fatigue strength of steel specimens is produced by the simultaneous action of a mildly corrosive liquid in contact with the steel while it is being repeatedly stressed. The resistance to this type of combined action (called corrosion-fatigue strength) depends chiefly on the ability of the surface material to resist localized corrosive attack; unless corrosion pitting is prevented, increasing the static strength and ordinary fatigue strength of a steel by alloying or heat treatment does not materially affect the corrosion-fatigue strength.

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A study of the available test data[2] leads to the conclusion that a significant increase in the corrosion-fatigue strength of a steel could be obtained if the surface were coated or otherwise treated in such manner that the localized corrosion pits usually developed by the simultaneous corrosion and repeated stressing would either be inhibited or prevented from progressive deepening with time.

TABLE I.—HEAT ANALYSES OF STEELS.

Steel	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Chromium, per cent	Vanadium, per cent	Silicon, per cent	Nickel, per cent
S.A.E. 1020......	0.20	0.42	0.009	0.026	....	....	....	....
S.A E. 3140......	0.37	0.71	0.015	0.020	0.61	....	0.21	1.40
S.A.E. 6120......	0.20	0.68	0.017	0.024	0.87	0.15	....	....

TABLE II.—PROTECTIVE COATINGS TESTED.

Type of Steel	Preliminary Heat Treatment	Protective Treatment	Type of Coating	Final Thickness of Coating, in.
S.A.E. 1020.......	Annealed at 1610 F.	Heated 1½ hr. at 1840 F. in mixture of silicon carbide and chlorine gas, cooled in furnace	Silicon impregnation	0.02
S.A.E. 3140.......	Normalized at 1600 F.; quenched in oil from 1510 F.; tempered at 1100 F.	Electrolytic bright zinc plating	Zinc	0.00015
		Electrolytic cadmium plating	Cadmium	0.0001
		2 coats air hardenable bakelite varnish	Bakelite varnish	...
		2 coats heat hardenable bakelite varnish, baked at 300 F.	Bakelite varnish	...
S.A.E. 3140.......	Normalized at 1650 F.	Heated in cyanide salt bath at 1520 F. for ½ hr., quenched in oil, tempered at 400 F.	Cyanide case	0.004
S.A.E. 6120.......	Quenched in oil from 1630 F.; tempered at 980 F.	Heated 60 hr. at 970 F. in ammonia gas	Nitrided case	0.02

TABLE III.—MECHANICAL PROPERTIES OF STEELS TESTED.

Material and Condition	Tensile Yield Strength (0.1% offset), psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Rockwell Hardness	Endurance Limit in Air, psi.
Silicon impregnated S.A.E. 1020 steel.......	26 000[a]	51 000	40.0[b]	42.3	Core B-81 Case B-99	22 500
Quenched and tempered S.A.E. 3140 steel...	93 850	124 150	20.0	68.5	C-21	67 000
Cyanided S.A.E. 3140 steel..................	181 000[a]	238 000	[c]	[c]	Core C-37 Case C-66[d]	...
Nitrided S.A.E. 6120 steel..................	103 750	122 150	...	[c]	Core C-22 Case C-57[d]	105 000

[a] One specimen. All other values are average of three tests.
[b] Specimen was 1 in. in diameter. All other specimens were 0.35 in. in diameter.
[c] Negligible.
[d] As converted from Vickers-Brinell readings.

The purpose of the investigation herein reported was to determine quantitatively the protection offered by several commercial impregnations and coatings on steel specimens subjected to completely reversed cycles of flexural stress while under the corrosive influence of a stream of fresh tap water. A comparison was made of the endurance limits of specimens tested in air with the endurance limits of coated specimens tested in water. The effects of thin electroplated zinc and cadmium coatings, heat and air-hardenable bakelite varnishes, cyaniding, nitriding, and silicon impregnation, on specimens subjected

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[2] For an extensive bibliography see H. J. Gough, "Corrosion-Fatigue of Metals," *Journal*, Inst. Metals, Vol. XLIX, No. 2, p. 17 (1932).

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to this type of corrosion-fatigue test were studied.

SPECIMENS, MATERIALS AND COATINGS TESTED

Tests were made on seven groups of specimens, each group of which had a different kind of protective coating, and three different steels were used in the tests: namely, S.A.E. 3140, S.A.E. 6120, and S.A.E. 1020. The heat analyses of the compositions of these steels are given in Table I, and a brief outline of the various heat treatments used and the methods of applying the protective coatings to specimens of each group, is shown in Table II. The mechanical properties of each steel in the final test condition are listed in Table III.

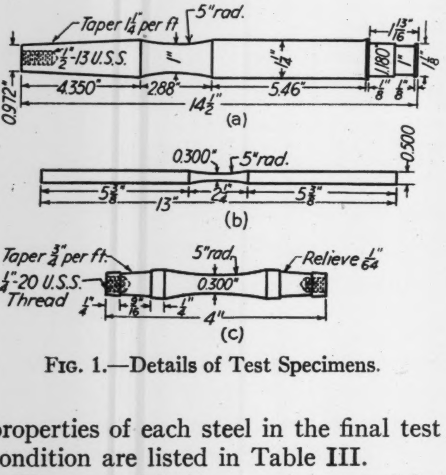

FIG. 1.—Details of Test Specimens.

The type of fatigue specimen used for the silicon impregnated coating is shown in Fig. 1 (*a*), and that for the nitrided and cyanided coatings is shown in Fig. 1 (*c*). The other fatigue specimens of the S.A.E. 3140 steel were all of the type shown in Fig. 1 (*b*). All groups of specimens except those with the silicon impregnated coating were given a final longitudinal polish with No. 00 emery paper before the protective coatings were applied and were not repolished before testing. The silicon impregnated case on the S.A.E. 1020 steel was approximately 0.03 in. thick after treatment and about 0.01 in. of this case was ground off to remove surface roughness from the final specimen. These specimens were rough polished before the silicon impregnation treatment and were subsequently ground and given the final longitudinal polish with No. 00 emery paper before testing.

Two types of machines were used in making these tests: (*a*) Farmer type rotating-beam machines in which the 0.30-in. diameter specimens were tested, and (*b*) rotating cantilever beam machines[3] in which the 1-in. diameter specimens were tested. Water was piped to the corrosion-fatigue machines directly from the supply mains and a small stream allowed to run on the specimens while they were being stressed. The total hardness of this water varies from 250 to 300 p.p.m., dissolved oxygen varies from 6 to 8 p.p.m., and the pH ranges from 7.8 to 8.2. The average temperature of the water during these tests was approximately 63 F., and each specimen was subjected to approximately 1450 reversals of stress per minute. All traces of oil were removed from the specimens tested in corrosion-fatigue by first washing the specimens in gasoline and then cleaning with acetone.

TEST DATA AND DISCUSSION OF RESULTS

Typical corrosion-fatigue failures of several specimens of S.A.E. 3140 steel are shown in Fig. 2. The number of corrosion-fatigue pits appearing on the surface of the coated or uncoated S.A.E. 3140 steel specimens was a function of the stress and type of coating. In general, for a given type of protective

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[3] For a complete description of the essential features of these machines see H. F. Moore and G. N. Krouse, "Repeated Stress (Fatigue) Testing Machines Used in the Materials Testing Laboratory of the University of Illinois," *Circular No. 23*, Engineering Experiment Station, University of Illinois (1934).

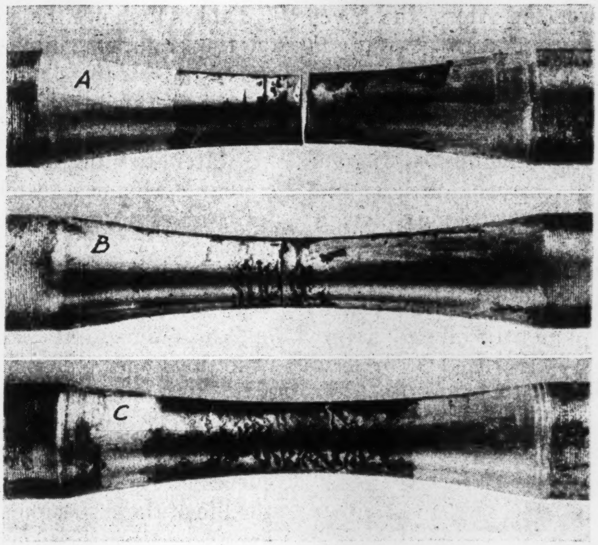

FIG. 2.—Corrosion-Fatigue Specimens of S.A.E. 3140 Steel After Failure.

(*A*) Cadmium plated; after 77 million cycles at 18,000 psi.
(*B*) Uncoated; after 102 million cycles at 15,000 psi.
(*C*) Zinc plated, after 108 million cycles at 30,000 psi.
(A light surface deposit from the water was removed by rubbing with fine emery paper before photographing.)

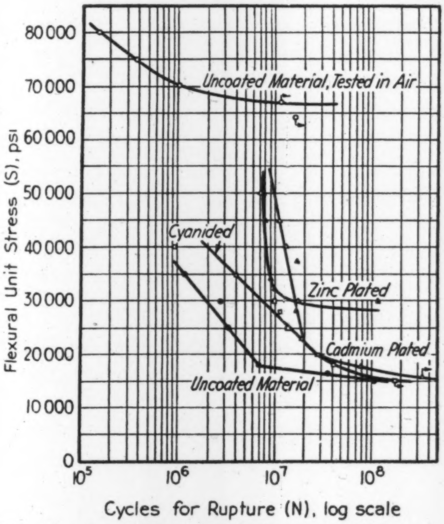

FIG. 3.—*S-N* Diagrams for Reversed Flexural Fatigue Tests of S.A.E. 3140 Steel. Tested in water unless noted otherwise.

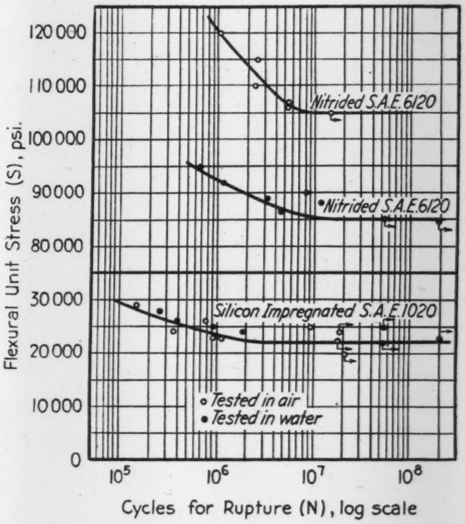

FIG. 4.—*S-N* Diagrams for Reversed Flexural Fatigue Tests of Silicon Impregnated S.A.E. 1020 and Nitrided S.A.E. 6120 Steel.

coating, the lower the stress, the fewer and deeper were the corrosion-fatigue pits when the specimen ruptured. Furthermore, the specimens with the zinc plating (which is electrochemically more active than cadmium) exhibited a larger number of small corrosion-fatigue pits than those with the cadmium plating. It is interesting to note that no visible corrosion pits were found on any of the nitrided or cyanided specimens tested; these coatings evidently offered a protection against localized corrosive pitting.

TABLE IV.—ENDURANCE LIMITS OF SPECIMENS TESTED.

Based on 100,000,000 cycles of stress.

Material	Endurance Limit in Air, psi.	Endurance Limit in Water, psi.	Ratio of Endurance Limit in Water to Endurance Limit in Air, per cent
Uncoated, S.A.E. 3140 Quenched and Tempered	67 000	15 000	22.4
Cadmium plated, S.A.E. 3140	[a]	17 000	25.4
Zinc plated, S.A.E. 3140	[a]	28 000	41.8
Cyanided, S.A.E. 3140	[a]	16 000	23.8
Nitrided, S.A.E. 6120	105 000	85 000	80.9
Silicon impregnated, S.A.E. 1020	22 500	22 500	100.0

[a] No data are available concerning the endurance limits of these specimens in air. As a basis on which to determine the percentage protection against corrosion offered by these coatings, the endurance limit in air of the quenched and tempered, uncoated material is used.

The *S-N* curves in Figs. 3 and 4 show the results obtained for each specimen tested, and the endurance limits for each type of coating were scaled from these curves as the stress which was just sufficient to cause failure in 100 million cycles of stress. Even though no definite horizontal asymptotes are obtained to the *S-N* diagrams for corrosion-fatigue tests, it was felt that this was a sufficiently large number of cycles of stress on which to base the relative protective value of various coatings.

Table IV shows the effect of corrosion on the endurance limits of the several steels and coatings tested. In the last column of this table are listed the ratios of the endurance limits of the specimens in water to the endurance limits of similar specimens tested in air, which ratio gives a measure of the relative efficiency of the protective coating in corrosion fatigue.

The results of corrosion-fatigue tests on cadmium- and zinc-plated S.A.E. 3140 steel specimens indicate that only a small amount of permanent protection against corrosive attack was produced by these coatings. From the *S-N* curves in Fig. 3 it may be observed that the life of these specimens was materially lengthened by the application of either coating if the part was subjected to cycles of stress above the corrosion-fatigue endurance limit. However, only a small increase in utilizable strength of the steel would result from the thin cadmium-plated coating when the part was subjected to an indefinitely large number of cycles of relatively low stress, whereas the thin zinc plating produced a definite increase in the corrosion-fatigue strength. Both of these electrolytically plated coatings were very thin and would usually be intended only for very mild service; perhaps thicker platings would produce a somewhat more effective protection.

The protective efficiencies of the electrolyticly plated coatings as judged by the ratios of the endurance limits when tested in water to the endurance limits in air (which ratios are listed in the last column of Table IV) were much smaller than those obtained by Harvey[4] in his study of zinc and cad-

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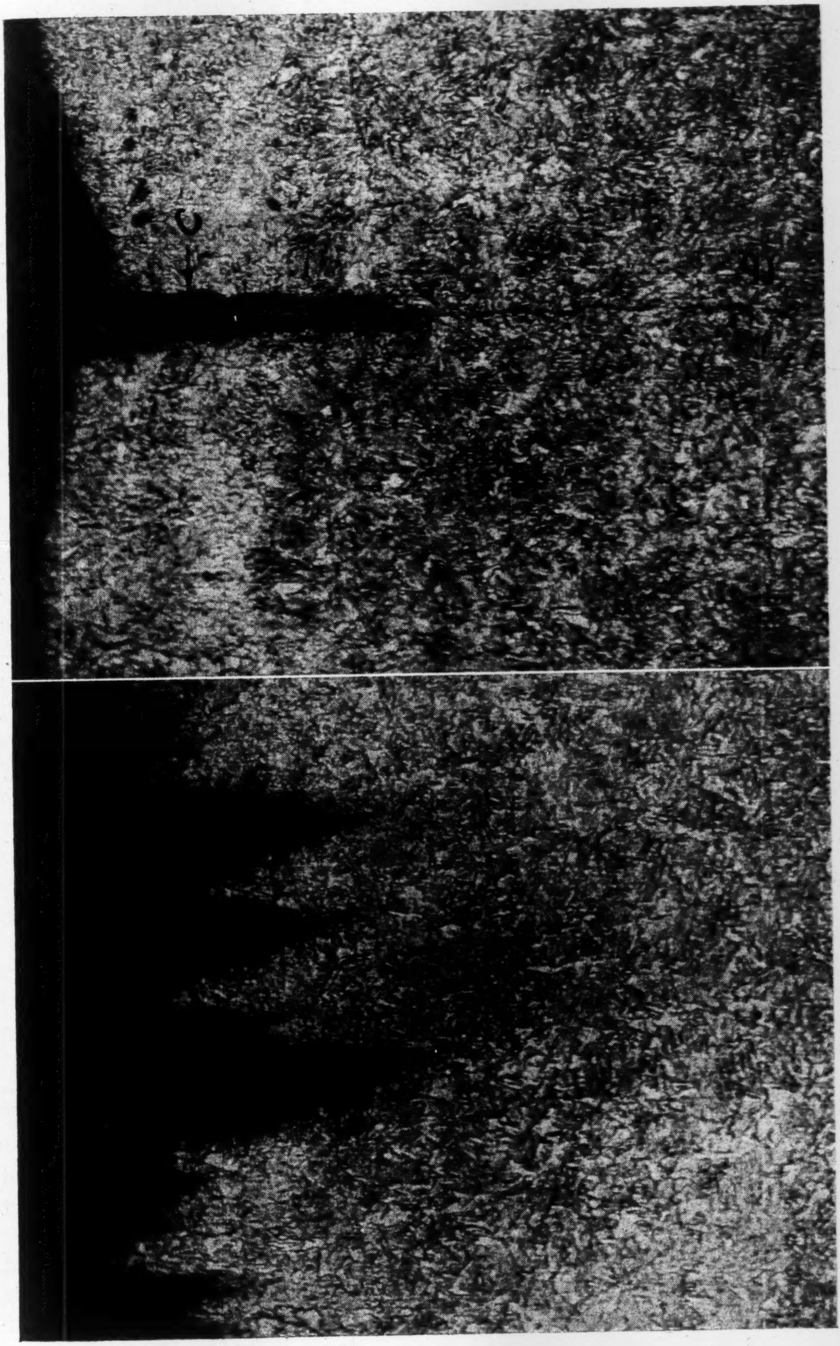
[4] W. E. Harvey, "Cadmium Plating *versus* Corrosion-Fatigue," *Metals and Alloys*, Vol. 3, No. 3, March, 1932, pp. 69 72; also "Zinc as a Protective Coating Against the Corrosion-Fatigue of Steel," *Metals and Alloys*, Vol. 1, No. 10, April, 1930, p. 458.



Zinc plated.

Cadmium plated.

FIG. 5.—Corrosion-Fatigue Pits in Plated Specimens of S.A.E. 3140 Steel (× 400).

Longitudinal section. Etched in 2 per cent Nital. Fatigue crack *A-B* developed from the corrosion pit *C*.

mium as protective coatings for corrosion-fatigue. This difference can probably be accounted for by the fact that his platings were much thicker, and he used a smaller number of cycles of stress as a criterion of the corrosion-fatigue endurance limit than those of the tests herein reported. There undoubtedly is a progressive lowering of the corrosion-fatigue endurance limit as the number of cycles of stress (and corresponding period of corrosion) are increased. After a corrosion pit has once formed in a coated flexural fatigue specimen, it is only a question of time before the pit develops to a sufficient depth to originate a final fatigue fracture. Pits of the types shown in Fig. 5 were developed on both cadmium- and zinc-coated specimens after several million cycles of stress while in contact with water.

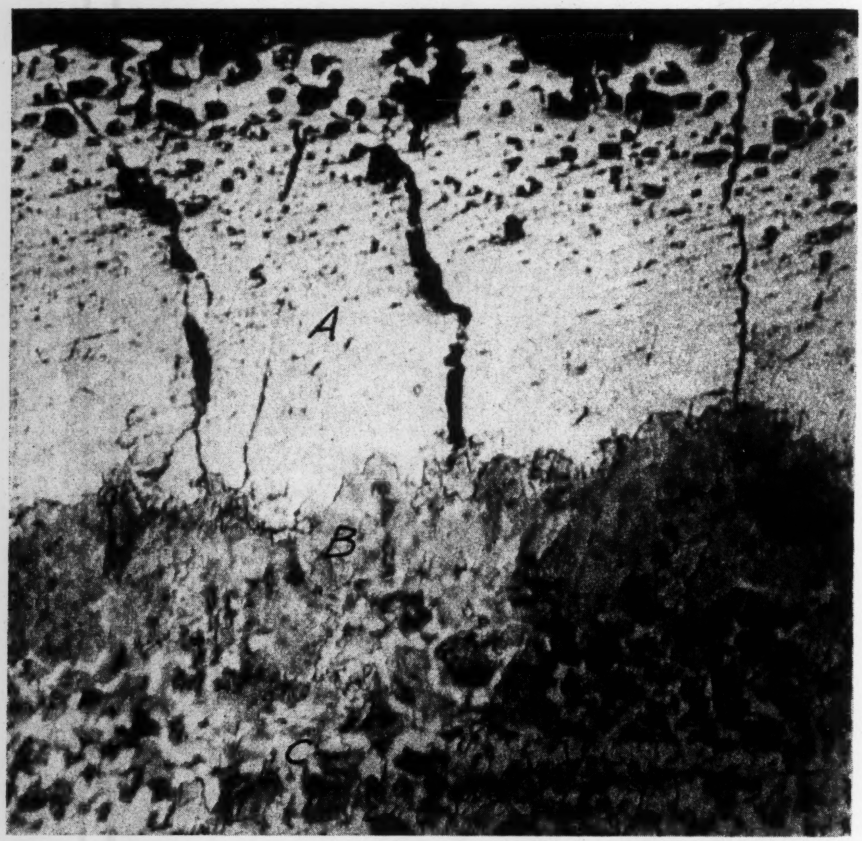


FIG. 6.—Fatigue Cracks in Silicon Impregnated Case on S.A.E. 1020 Steel (× 100).

Longitudinal section of specimen. Etched in 2 per cent Nital. The case *A*, has a high silicon content. The carbon was displaced from the area of the case and formed a layer *B* of high carbon content beneath the case. The material in the core (below *C*) was a pearlitic low carbon steel

Cyaniding of the S.A.E. 3140 steel did not produce a definite increase in corrosion-fatigue strength of the specimens tested. While the specimens showed no visible signs of corrosion pitting, which would indicate a protection against corrosive action, it was evident that the carbon content of the original steel was too high or perhaps the treatment employed was unsatisfactory for proper application of a cyanide case. The specimens were extremely brittle and warped slightly in quenching. Possibly some residual stresses were also present in the specimens and the combined effect of these undesirable conditions was to lower the endurance limit very materially. Perhaps a cyanide case would be effective in increasing the corrosion-fatigue strength of a low-carbon steel if the treatment could be properly controlled.

While no data are available concerning the endurance limit in air for the untreated S.A.E. 6120 steel, it can be assumed from the high endurance limit obtained (105,000 psi.) that the nitrided surface on this steel greatly increased the fatigue strength of the specimens in air. The data in Table IV indicate that the nitrided case was very effective in resisting the corrosive attack and that approximately 80 per cent of the strength of the nitrided steel, as determined by the endurance limit of the material in air, can be utilized if the material is simultaneously subjected to fresh water corrosion and fatigue. The endurance limit of 85,000 psi. obtained for the nitrided specimens tested in water is higher than that obtained for specimens of most steels when tested in air and is about four times as great as the usual corrosion-fatigue endurance limits of most steels.

The silicon impregnated S.A.E. 1020 steel was the only material studied that had a corrosion-fatigue endurance limit equal to the endurance limit in air. The fatigue strength of the S.A.E. 1020 steel in air was probably lowered somewhat by the application of the high silicon case; however, the corrosion-fatigue endurance limit of untreated S.A.E. 1020 steel would normally be lower than that obtained for the specimens with the silicon impregnated case. One factor which affected the strength of the silicon impregnated, low-carbon steel was the porosity of the case which developed a notch effect on the specimens subjected to repeated loading. The photomicrograph in Fig. 6 shows several fatigue cracks which progressed through the case of the material and in some instances started from pits in this porous surface. The strength of these silicon impregnated specimens in fatigue would normally be governed by the strength of the case and the notch-sensitivity of the material; perhaps the fatigue strength could be improved somewhat by close metallurgical control to prevent formation of deep pits, and by grinding off the case to a greater depth to remove most of the outer porous layer of the material. No corrosion pits were observed on the surface of the silicon impregnated specimens that were tested in water; even after two hundred million cycles of a stress in the neighborhood of the endurance limit only a slight discoloration of the surface could be detected.

TABLE V.—RESULTS OF CORROSION-FATIGUE TESTS OF S.A.E. 3140 STEEL COATED WITH BAKELITE VARNISHES.

Flexural Stress, psi.	Cycles to Fracture
Heat Hardenable Bakelite Varnish XV 11444	
40 000	2 628 300
30 000	171 122 000[a]
Air Hardenable Bakelite Varnish 15BK	
50 000	5 780 100
40 000	164 890 000[a]
Air Hardenable Bakelite Varnish 12BK	
40 000	14 497 800
30 000	13 488 700

[a] Specimen did not fracture, but test was stopped at the number of cycles indicated.

Only a few tests were made of specimens with surfaces coated with bakelite varnishes and the results for each specimen tested are shown in Table V. By comparison with the *S-N* curves in Fig. 3, it will be noted that all of these bakelite varnishes were effective in increasing the life of the specimen beyond that of the untreated steel, and that two of these varnishes apparently increased the corrosion-fatigue endurance limits to values above those obtained for any of the other coatings employed on the S.A.E. 3140 steel. It was found difficult to obtain a good uniform adherence of the varnish without the necessity of roughening the surfaces of the specimens, and the fractured specimens showed an irregular distribution of corrosion pitting at localized areas in which the varnished surface had dust particles imbedded or had cracked at random when stressed during the test. For service conditions in which abrasion is not likely to occur, some of the bakelite varnishes may offer an effective means of protecting a member from localized corrosive attack and should materially increase the corrosion-fatigue strength of a member if an impervious adherent coating is obtained.

CONCLUSIONS

In general, the corrosion-fatigue endurance limits for specimens with commercially used protective coatings were considerably below the endurance limits of the steels tested in air. Specimens with a silicon impregnation case exhibited the same endurance limit when tested in air or in water, but the value obtained for this endurance limit was comparatively low (about 22,500 psi.). Zinc-plated specimens had a greater corrosion-fatigue strength than the cadmium-plated specimens, but both platings were rather thin and gave endurance limits less than 45 per cent of the fatigue strength of the uncoated steel in air. Specimens with a nitrided case were very efficient in resisting pitting by corrosive attack, and a very high corrosion-fatigue strength was obtained for the nitrided specimens (85,000 psi.).

Acknowledgment:

This investigation was conducted as a part of the work of the Engineering Experiment Station of the University of Illinois, of which M. L. Enger is the director, and of the Department of Theoretical and Applied Mechanics, of which F. B. Seely is the head.

The cooperation of the following companies in treating or plating the various specimens is gratefully acknowledged:

Lindberg Steel Treating Co., Chicago, Ill.
Link Belt Co., Dodge Plant, Indianapolis, Ind.
Globe Steel Tubes Co., Milwaukee, Wis.

The cooperation of the Barrett Varnish Co. and of the Bakelite Corporation of America in furnishing the varnishes tested is greatly appreciated.

DISCUSSION

Mr. Blaine B. Wescott[1] (*presented in written form*).—The authors have presented some very interesting data pertaining to the protective value of various types of electroplated coatings and surface treatments against corrosion-fatigue. The tests made on the steels with the different surface treatments, silicon impregnation, cyanide case, and nitrided case, provide useful information, but the same is not true of the tests made to evaluate the electroplated zinc and cadmium coatings. Although the thin electroplated coatings perhaps may be representative of some commercial applications for temporary protection against corrosion they definitely could not be considered to have any worthwhile possibilities for protection against corrosion-fatigue. Undoubtedly, tests would have shown the presence of numerous "holidays" or pinholes in these coatings, and such a condition is known to be not conducive to resistance to corrosion-fatigue. The value of the data would have been increased greatly if these tests had been made on impervious electroplated coatings of appreciable thickness.

TABLE I.—CORROSION-FATIGUE LIMITS OF STEELS WITH ELECTROPLATED COATINGS.

Type of Steel and Treatment	Type and Thickness of Coating, in.	Corrosion-Fatigue Limit, psi.		Damage Ratio[a]
		In Air	In Sulfide Brine	
0.38% C, 1.91% Mn—normalized 1600 F.	none	56 400	12 100	0.21
	bright zinc – 0.0005		19 800	0.35
	bright zinc – 0.0010		22 900	0.41
	ductile zinc 0.0005		19 100	0.34
	ductile zinc 0.0010		19 900	0.35
S.A.E. 4620—normalized 1650 F., drawn 1050 F.	none	52 500	22 500	0.43
	nickel – 0.005	40 000	34 900	0.67
0.40% C, 0.20% Cu	none	41 100	11 000	0.27
	zinc – 0.0023		25 100	0.61

[a] Damage ratio is ratio of sulfide corrosion-fatigue limit to air-fatigue limit of uncoated steel.

The data in the accompanying Table I are offered as additional information on the utility of electroplated metallic coatings for protection against corrosion-fatigue. The tests were made on R. R. Moore machines modified so that the samples were completely immersed in the corrosive medium in the absence of air. These conditions were selected so as to approach the service conditions as closely as possible. The corrosive medium was an oil-well brine saturated with hydrogen sulfide. The details of the test method and the composition of the brine have been published elsewhere.[2] The corrosion-fatigue limits were determined for 10,000,000 reversals of stress so that the values are merely comparative and not necessarily indicative of the life of the coatings.

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[1] Engineer of Tests, Gulf Research and Development Co., Pittsburgh, Pa.

[2] B. B. Wescott and C. N. Bowers, "Corrosion Fatigue and Sucker Rod Failures," Am. Petroleum Inst. Fourteenth Annual Meeting, Chicago, Ill., October 24, 1933: *Production Bulletin No. 212* (1933).

These results indicated that the protective value is proportional roughly to the coating thickness. Examination of the samples indicated that failure followed rupture of the coating rather than deterioration through corrosion. On this basis, if any worth-while advantage is to be gained from electroplated coatings it is essential that the coating itself and the bond to the base metal be capable of withstanding the deformation caused by the applied service stresses. Failure in this respect will nullify the corrosion resistance of the coating.

In the one case where the air endurance limit of the steel uncoated and coated could be determined, it was found that nickel plating caused a reduction of 24 per cent. The thicker coatings of both nickel and zinc materially increased the corrosion-fatigue limit under the conditions of testing. The fact that the percentage increase was so much greater for the zinc coating than for the nickel coating nearly twice as thick suggests that it would be both interesting and useful to study the magnitude of the electrochemical effects. It is hoped through further tests to develop more definite data relative to the effective life of electroplated nickel and zinc coatings.

MESSRS. THOMAS J. DOLAN[3] AND HUGH H. BENNINGER[3] (*Authors' closure by letter*).—It was pointed out in the paper, and Mr. Wescott has emphasized the fact, that the electroplated zinc and cadmium coatings on the authors' specimens were very thin and would usually be intended only for mild service conditions. The authors agree that an increased thickness of coating probably would offer an increased corrosion-fatigue limit in laboratory tests based on a relatively short corrosion time, but there is always a question as to permanence of such protective effect when a member is subjected to this particularly damaging service condition during a long operating life.

Some further corrosion-fatigue data on electrolytic lead and cadmium platings 0.0005 in. thick, on specimens of the same heat of quenched and tempered S.A.E. 3140 steel as that reported in the paper, have been obtained by the authors since the original paper was presented. The authors wish to express their appreciation to the engineers of the Chrysler Corp. who cooperate in plating these specimens. The *S-N* curves for these specimens are designated as *A* and *C* in the accompanying Fig. 1, and for comparison the curve marked *B* shows the data (originally plotted in Fig. 3 of the

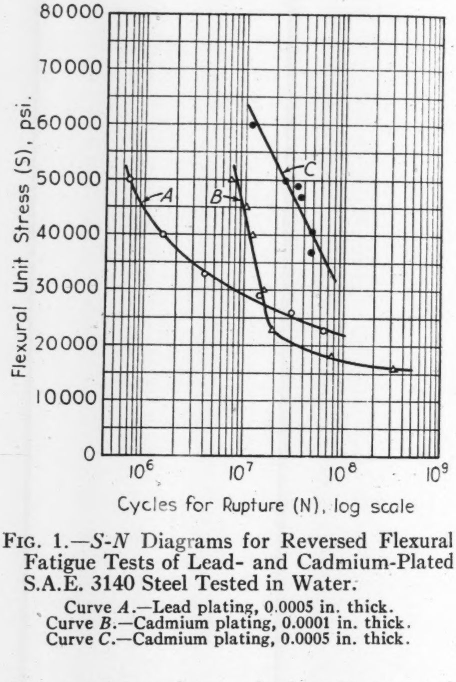

FIG. 1.—*S-N* Diagrams for Reversed Flexural Fatigue Tests of Lead- and Cadmium-Plated S.A.E. 3140 Steel Tested in Water.

Curve *A*.—Lead plating, 0.0005 in. thick.
Curve *B*.—Cadmium plating, 0.0001 in. thick.
Curve *C*.—Cadmium plating, 0.0005 in. thick.

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[3] Assistant Professor of Theoretical and Applied Mechanics, and Former Graduate Student, respectively, University of Illinois, Urbana, Ill.

paper) for the specimens with a cadmium plating 0.0001 in. thick. The corrosion-fatigue endurance limit obtained for the lead-plated specimens was about 22,000 psi., but unfortunately there were not sufficient specimens available to obtain the corrosion-fatigue endurance limit of the specimens with the thick cadmium plating. An arbitrary extrapolation of curve *C* would indicate a probable endurance limit of about 30,000 psi. at 100 million cycles of stress. Both of these values appear low when contrasted with the endurance limit of 67,000 psi. for the uncoated S.A.E. 3140 steel tested in air.

The specimens with the thicker cadmium plating (curve *C*) did show a much greater life before fracture, and exhibited a greater endurance limit based on 10 million cycles of stress for fracture than did the thin cadmium plated specimens (curve *B*). However if the endurance limit corresponding to several hundred million cycles of stress had been determined for the thicker cadmium plating, a much smaller difference in corrosion-fatigue strengths for these two thicknesses of coating probably would have been obtained.

A comparison of curves *A* and *B* in Fig. 1 indicates the danger involved in drawing conclusions from corrosion-fatigue tests based on relatively few cycles of stress or relatively short corrosion time. By scaling the ordinates at 10 million cycles of stress the data show that the thin cadmium-plated specimens (curve *B*) had an endurance limit of 44,000 psi. and the lead-plated specimens (curve *A*) had an endurance limit of only 29,000 psi. Thus the thin cadmium-plated specimen had an endurance limit much *greater* than that of the specimens having a lead plating five times as thick. However, if a comparison of the corrosion-fatigue strengths is made at a much greater number of cycles of stress (say 50 to 100 million) the cadmium-plated specimens have an endurance limit about 5000 psi. *less* than that of the lead-plated specimens.

Mr. Wescott reports data showing that a zinc coating gave a greater percentage of increase in corrosion-fatigue strength than a nickel coating that was nearly twice as thick. Since his data are based on tests run to only 10 million cycles of stress and compare results of tests on specimens of two different steels, the relative corrosion-fatigue strengths of the two coatings might be quite different if the tests were continued and a comparison made at about 100 million cycles of stress on specimens of the same steel.

As Mr. Wescott has pointed out, it is important to observe that the corrosion-fatigue strength of a metal member cannot be improved by coating or plating unless the fatigue strength of the coating material is sufficient to withstand the deformations caused by the repeated service stresses without cracking or flaking off. Even though a coating has a great resistance to corrosion, it will not be an effective protective surface if small pinholes or cracks allow the corrosive medium to gain access to the base metal and thereby initiate corrosion-fatigue cracks at localized points. In laboratory corrosion-fatigue tests the two damaging factors—repeated stress and corrosion—are interacting to varying degrees to control the life of a specimen. Perhaps in testing plated specimens at relatively high stresses the resistance of the plating to fatigue cracking is the dominant factor controlling the life of a specimen, whereas at lower stresses the relative corrosion resistance of the coating may be the governing factor that determines how long a specimen will run before corrosion-fatigue cracks progress into the base metal.

SOME OBSERVATIONS OF THE POTENTIALS OF METALS AND ALLOYS IN SEA WATER*

By F. L. LaQue[1] and G. L. Cox[1]

Synopsis

This paper comprises a review of the literature on the potentials of metals and alloys in sea water and similar chloride solutions. It also contains a description of a particular method of determining potentials of a number of metals and alloys by the use of specimens in the form of pipe through which sea water was circulated during the test period of about six months. The paper also includes results of previous tests made in a similar manner, and other tests of galvanic couples in sea water under natural conditions.

All of the data are used in setting up a galvanic series referring to sea water which indicates the relative positions of about forty common metals and alloys. The data are shown graphically and in tables. The apparatus used is illustrated by a diagram and a photograph of the test setup.

The program of Subcommittee VIII on Galvanic and Electrolytic Corrosion of the Society's Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys has, for some time, included a study of the galvanic corrosion of certain metals and alloys when completely immersed in sea water. The couples to be studied included all combinations of copper, lead, brass, tin, bronze, monel,[2] copper-nickel, and 18-8 stainless steel, plus all combinations of copper, lead, aluminum, iron, nickel, tin, and zinc without duplicating the copper-lead couples in the second series.

In pursuance of this program, some preliminary tests on typical couples have been carried out with the principal object of determining the practicability of the proposed method of test. Further information concerning these preliminary tests will be found in the reports of Subcommittee VIII. This preliminary work has established that the proposed procedure was adequate and practical, although if the program were to be put into effect, it would be desirable to make a few minor changes in the design of the specimens and racks and to re-examine the list of couples to be tested with the object of eliminating some and adding others. These preliminary tests also provided a basis for estimating the cost of completing the proposed program.

In considering this proposed work from the standpoint of cost in both time and money, it occurred to Subcommittee VIII that it might be well to review the data on the same subject which have become available since the program was drawn up several years ago. There was a strong possibility that, in the light of data already available, the proposed tests would serve merely to confirm what was already sufficiently well established, and that, in consequence, the efforts of the committee might be directed more profitably to other aspects of the subject of galvanic corrosion in sea water.

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* The presentation of this paper at the annual meeting was sponsored by Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.

[1] Research and Development Division, The International Nickel Co., Inc., New York, N. Y.

[2] Registered at U. S. Patent Office.

PURPOSE

The purpose of the present paper is to report some results of tests of galvanic couples in sea water that have been carried out by the authors and their associates during the past few years, and to compare these results with some data pertaining to the same subject that have been reported by others. It is believed that these data, when qualified and augmented by critical discussion by other investigators, will be as generally useful as the information that might be derived from the proposed testing program.

METHODS OF STUDYING GALVANIC CORROSION

The results of no galvanic corrosion test can ever be more than semiquantitative. A principal reason for this is that the magnitude of the galvanic effect in any case is determined to a very important extent by the ratio of exposed areas of the metals forming the couple. Consequently, the quantitative results of any single test may be applied directly only to combinations of the same materials in which the same ratio of areas exists, and where the conditions of exposure are otherwise identical with those which obtained during the test.

The effects of relative areas of anode and cathode have been studied by several investigators (**1, 2, 3**),[3] and some observations referring particularly to the iron-copper couple in sodium chloride are to be reported in a companion paper by W. A. Wesley.[4]

The principal information likely to be derived from galvanic corrosion tests is the direction of current flow in the galvanic circuits. The extent of the galvanic effects will be influenced by the relative areas of anode and cathode, the polarization characteristics of the metals in the corroding solution, the internal and external resistances in the galvanic circuit, the potential developed by the couple, as well as the several factors that normally influence corrosion processes.

The principal means of studying the galvanic behavior of metals and alloys are:

1. Measurement of the open-circuit potential difference between metals or alloys forming a couple.
2. Measurement of the potential of a metal or alloy in the testing solution with reference to a standard of potential such as a calomel half cell.
3. Measurement of the direction and value of the current generated by a couple throughout a test period.
4. Comparison of the extent of corrosion of coupled and uncoupled specimens exposed under identical conditions. Such measurements of the extent of corrosion may be made by means of weight-loss determinations, observations of changes in mechanical properties (strength and ductility), and measurements of the extent and distribution of local attack (pitting).
5. Determination of the polarization characteristics of the specimens in the testing solution.

Open-circuit potential measurements, including those referred to a standard half cell, do not indicate the effects of continued current flow, but do provide a means of following changes in the electrode behavior of specimens and of observing fluctuations in relative potential differences among the members of a series of metals and alloys.

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p 687

[4] See p. 690

Potential measurements also provide a means of detecting either local or general changes in the characteristics or state of repair of protective or passive films on metal surfaces. Such effects are influenced greatly by the polarization characteristics of the metal and of the solution. Brown and Mears (3) have shown that with aluminum in 0.1 *N* potassium chloride, which gave no appreciable anodic polarization, the activation of even an extremely small portion of the surface was followed by a sharp and considerable increase in potential of the specimen in the direction of lower nobility. Similar experiments by these investigators (4), using stainless steel specimens in dilute sodium chloride, showed a generally similar behavior, although in this case there was some evidence of anodic polarization. When ferric chloride, a powerful cathodic depolarizer, was added to the dilute sodium chloride used in the experiments with stainless steel, the effect of local activation on the observed potential of the stainless steel specimen was suppressed to a very considerable extent. This observation led these investigators to the conclusion that it was undesirable to use single potential measurements to detect initial corrosion breakdowns in cases where the polarization characteristics of the metal and solution are not understood.

In the investigation to be described here, it was discovered that the potentials of a number of stainless steel specimens fluctuated to a very considerable extent, and there were considerable differences in potential between apparently identical specimens from the same lot of material. This behavior would be consistent with what might be expected under exposure conditions such as probably pertained to this investigation. The corrosion reaction would be under cathodic polarization control with little anodic polarization and, therefore, the incidence or cessation of local corrosive attack would be reflected in a considerable change in the observed potential of a specimen.

TABLE I.—ELECTROMOTIVE SERIES.

Metal	Molal Electrode Potential at 25 C.	
	Ion	Volts
Magnesium	Mg^{++}	−2.34
Aluminum	Al^{+++}	−1.67
Zinc	Zn^{++}	−0.76
Chromium	Cr^{+++}	−0.71
Iron	Fe^{++}	−0.44
Cadmium	Cd^{++}	−0.40
Nickel	Ni^{++}	−0.25
Tin	Sn^{++}	−0.14
Lead	Pb^{++}	−0.13
Hydrogen	H^{+}	arbitrary zero point
Copper	Cu^{++}	+0.34
Silver	Ag^{+}	+0.80
Palladium	Pd^{++}	+0.83
Mercury	Hg^{++}	+0.85
Platinum	Pt^{++}	+1.2
Gold	Au^{+++}	+1.42

Potential measurements supplemented by frequent determinations of the magnitude and direction of current flow provide considerably more information than can be derived solely from weight-loss determinations and physical tests made after exposure, which measure only the net effect of coupling without disclosing much else about the characteristics of the elements of the couple.

Data will be presented as determined by each of the methods mentioned. In each case, these data will be used only to establish the positions of the metals and alloys in a galvanic series referred to salt water. Numerical data will be presented merely to facilitate comparison between the results of different investigators and to indicate the relative potential differences between different materials more clearly than by a simple listing. Because of the effects of the incidental conditions of exposure, it is not proper to assign absolute values to any particular potential measurements.

TABLE II.—DATA FROM SEVERAL SOURCES ON THE POTENTIALS OF SOME METALS AND ALLOYS IN SALT WATER AND SIMILAR SOLUTIONS.
Potential in volts referred to saturated calomel half cell

Material	Reference[a]									
		B					F			
	A	Under Nitrogen	Under Oxygen	C	D	E	After Treatment with HCl	After Exposure to Air	G	H
Magnesium alloy DTD 88	..	..	..	−1.51	..	..	..	..	..	..
Zinc	..	−1.08	−1.07	−1.09	..	..	..	..	..	..
Cadmium	..	−0.81	−0.74	−0.78	−0.74 to −0.84	−0.73	..	..	..	..
Aluminum	..	−0.78	−0.77	<−0.78	..	..	..	..	..	..
Aluminum 52SH	..	..	..	..	..	−0.77	..	..	..	..
Aluminum 4S	..	..	..	..	..	−0.75	..	..	..	..
Aluminum 2S	..	..	..	..	..	−0.74	..	..	..	..
Aluminum 3S	..	..	..	..	..	−0.74	..	..	..	..
Aluminum 53S-T	..	..	..	..	..	−0.74	..	..	..	..
Alclad	..	..	..	..	..	−0.74	..	..	..	..
Aluminum A17S-T	..	..	..	..	..	−0.64	..	..	..	..
Duralumin	..	..	..	−0.61	..	..	..	..	..	..
Aluminum 17S-T	..	..	..	..	..	−0.59	..	..	..	..
Aluminum 24S-T	..	..	..	..	..	−0.59	..	..	..	..
Mild steel or iron	−0.75	−0.72	−0.65	..	−0.56 to −0.78	−0.40	−0.76	−0.71	..	..
Lead	..	−0.54	−0.51	..	−0.50	..	..	..	..	..
Tin	..	..	..	..	..	..	..	..	−0.46	−0.48
Nickel	..	−0.35	−0.26	..	..	..	−0.48	−0.24	..	..
Brass 3B12	..	..	..	−0.23	..	..	..	..	..	..
Naval brass	..	..	..	..	..	−0.19	..	..	..	..
Copper	..	−0.26	−0.21	..	−0.24	..	..	..	..	..
Aluminum bronze	..	..	..	−0.22	..	..	..	..	..	..
Gun metal	..	..	..	−0.17	..	..	..	..	..	..
Monel	..	..	..	−0.14	..	−0.01	..	..	..	..
18-8 stainless steel (type 304)	..	..	..	−0.11 to −0.21	..	−0.00	−0.54	−0.31[b]	..	..
18-8 stainless steel (type 316)	..	..	..	..	..	..	−0.53	+0.05[b]	..	..
Silver	..	−0.049	−0.055	..	..	..	..	..	..	..
Gold	..	+0.180	+0.175	..	..	..	..	..	..	..
Platinum	..	..	..	..	..	..	+0.30	+0.33	..	..

[a] The letters used in the columns of this table represent the following:
A - Bauer (6). Tests were made in 1 per cent sodium chloride for 100 hr.
B—Krueger and Kahlenberg (7). Tests were made in 1*N* potassium chloride under an atmosphere of either nitrogen or oxygen at 25 C.
C—Willstrop (8). Specimens were exposed to sea water at 23 C. in an open vessel without agitation.
D—Gatty and Spooner (9). Value for lead refers to test in molal potassium chloride at 25 C.
Value for copper refers to test in molal sodium chloride at 18 C.
Value for iron refers to test in molal potassium chloride at 25 C.
Value for cadmium refers to test in molal potassium chloride at 18 C.
E—Whitmore and Teres (10). Tests were made in molal sodium chloride which contained 0.3 per cent hydrogen peroxide at 23 C.
F—Uhlig (11). Tests on nickel, iron, and platinum were made in oxygen free 4 per cent sodium chloride at 25 C. after treatment with 6*N* HCl and after exposure to air. The values for the stainless steels refer to similar treatment with 6*N* HCl and subsequent exposure to air-saturated 4 per cent NaCl. A specimen of 18-8 stainless steel, type 304, exposed to air-saturated 4 per cent NaCl without previous activation reached a final passive potential of +0.09 v.
G—Brennert (12). Tests were made in molal sodium chloride at 25 C.
H—Hoar (13). Tests were made in molal potassium chloride at 25 C. Lower concentrations gave lower values and higher concentrations higher values, for example, from —0.31 v. in 0.001 molal to —0.54 v. in 4 molal potassium chloride.

[b] These values refer to tests in air saturated 4 per cent NaCl.

DATA FROM THE LITERATURE

To facilitate direct comparison, all potential values will be referred to the standard saturated calomel half cell. The values, referred to the standard hydrogen electrode at 25 C. for the different calomel half cells, used in recalculating the results of other investigators were:

Saturated calomel half cell........	+0.2458 v.
Normal calomel half cell..........	+0.2848 v.
One-tenth normal calomel half cell.	+0.3376 v.

For convenient reference, the standard molal potentials of some common metals are given in Table I. This table is based on the latest information as reported by Latimer (5). The values reported refer, of course, to the metals when they are in contact with a solution in which the activity of the ion indicated is one mole in 1000 g. of water.

TABLE IV.—POTENTIAL MEASUREMENTS BY GELLER (17) AFTER COUPLING ALUMINUM WITH OTHER MATERIALS IN 3 PER CENT SODIUM CHLORIDE AT 20 C.

Material	Potential, v. Referred to Saturated Calomel Half Cell 5 min. After Contact with Aluminum for 71 days	Potential, v. Referred to Saturated Calomel Half Cell of Aluminum Specimen in Couple
Duralumin.............	−0.754	−0.920
Ingot iron..............	−0.639	−0.871
15% Cr steel...........	−0.418	−0.872
18% Cr—1.8% Mo steel.	−0.287	−0.868
18% Cr—8% Mn steel...	−0.281	−0.881
15% Cr—8% Mn—1.5% Ni steel............	−0.254	−0.884
18% Cr—8% Ni steel..	−0.254	−0.872
Copper................	−0.231	−0.861

Some data from the literature referring to the potentials of metals and alloys in salt water and similar solutions are given in Table II. The materials have been arranged roughly in the form of an electromotive series.

Additional data on the galvanic relationships among metals and alloys in sodium chloride solutions have been determined by Edwards and Taylor (14), particularly with reference to aluminum and its alloys, and by Evans (15) on a number of other materials. Noyes (16) has reported similar data for specimens exposed to brackish water. Electromotive series based on these data are shown in Table III.

TABLE III.—GALVANIC SERIES.

Edwards and Taylor		Evans		Noyes	
Material	Calculated Potenial Difference, v.	Material	Calculated Potential Difference, v.	Material	Calculated Potential Difference, v.
Zinc	0.058	Zinc	0.15	Aluminum brass	0.021
Galvanized steel	0.280	Aluminum	0.14	Red brass	
99.2% aluminum	0.016	Cadmium	0.12	Copper	
51S-T aluminum	0.010	Lead	0.02	85-15 Cu-Ni	0.014
99.95% aluminum	0.023	Steel	0.11	70-30 Cu-Ni	0.051
Blue annealed steel	0.013	Tin	0.31	Comp. M-bronze	0.017
25ST aluminum	0.028	Nickel	0.10	Comp. G-bronze	0.158
Ingot iron	0.036	Copper		Monel	0.052
17S-T aluminum	0.381			18-8 stainless steel	
Brass (60-40)	0.007				
Copper					

Conditions of Test

Edwards and Taylor (14). Based on values obtained from tests in molal sodium chloride at atmospheric temperature.

Evans (15). Based on values obtained 7½ min. after immersion in 0.1 molal sodium chloride at atmospheric temperature.

Noyes (16). Plus private communication from U. S. Naval Engineering Experiment Station, Annapolis, Md. Based on values observed 30 min. after specimens in couples had been on closed circuit for several days with specimens being rotated at 30 r.p.m. in brackish Severn River Water at atmospheric temperature.

Geller (17) recently reported results of an investigation in which aluminum (99.5 per cent) was coupled with other materials in 3 per cent sodium chloride solution at 20 C. for various periods up to 71 days, after which the circuits were broken and the potentials of the members of the couples were determined after from 1 to 5 min. The data in Table IV refer to the values obtained after 5 min. on open circuit in each case.

The agreement among the results of the several investigations must be considered as surprisingly good in view of the difference in testing conditions and the naturally inconsistent behavior of materials that may assume varying degrees of passivity.

These data may be compared with the experimental data to be reported here, and will serve as part of the basis for the galvanic series in salt water which will be set up as the principal conclusion of this paper.

TABLE V.—NOMINAL COMPOSITIONS OF PIPE SPECIMENS INCLUDED IN GALVANIC TESTS.

Material	Conditions	Nickel, per cent	Copper, per cent	Chromium, per cent	Iron, per cent	Manganese, per cent	Carbon, per cent	Silicon, per cent	Molybdenum, per cent	Zinc, per cent	Tin, per cent	Aluminum, per cent	Lead, per cent
Galvanized wrought iron	Welded and galvanized	...	...	...	...	...	...	...	...	Std.	...	...	...
Cast iron	As cast	...	...	...	bal.	...	3.2	...	...	...	...	...	...
Mild steel	Welded	...	...	...	bal.	0.60[c]	0.25[c]	...	...	...	...	...	...
Ni-Resist[a]	As cast	14	6	2	bal.	1.5	3	1.7	...	...	...	...	...
Stainless steel, type 304	Cold drawn, seamless, annealed	8	...	18	bal.	...	0.08[c]	...	...	...	...	...	...
Stainless steel, type 316	Cold drawn, seamless, annealed and pickled	10	...	18	bal.	...	0.10[c]	...	2.5	...	...	...	...
Inconel[a]	Pickled, cold drawn, seamless	79.5	0.2	13	6.5	0.25	0.08	0.25	...	...	...	...	...
Nickel	Pickled, cold drawn, seamless	99.4[b]	0.1	...	0.15	0.2	0.1	0.05	...	...	...	...	...
Monel[a]	Pickled, cold drawn, seamless	67	30	...	1.4	1.0	0.15	0.1	...	...	...	...	...
70-30 copper-nickel	Pickled, cold drawn, seamless	30	70	...	...	...	...	...	...	...	...	...	...
Ambrac	Pickled, cold drawn, seamless	20	75	...	...	...	...	...	...	5	...	...	...
Comp. M-bronze	Cast and machined	...	88	...	...	...	...	...	...	4	6.5	...	1.5
Comp. G-bronze	Cast and machined	...	88	...	...	...	...	...	...	2	10	...	...
Manganese bronze	Cast and machined	...	58	...	1	1	...	...	...	39	...	1	...
Admiralty brass	Pickled, cold drawn, seamless	...	70	...	...	...	...	...	...	29	1	...	...
Red brass	Pickled, cold drawn, seamless	...	85	...	...	...	...	...	...	15	...	...	...
Yellow brass	Pickled, cold drawn, seamless	...	63	...	...	...	...	...	...	37	...	...	...
Silicon bronze	Pickled, cold drawn, seamless	...	98	...	...	0.25	...	1.5	...	...	...	...	...
Copper	Pickled, cold drawn, seamless	...	99.9+	...	...	...	...	...	...	...	...	...	...
Lead	Standard pipe	Chemical Lead											

[a] Registered at U. S. Patent Office.
[b] Including cobalt.
[c] Maximum.

PIPE TEST SETUP

After experiments with several methods of studying galvanic corrosion in sea water, the authors have directed their attention particularly to a testing apparatus to be described in detail. The other methods that have been used will be referred to only briefly, although the results obtained with these other methods will be used as supplementary data in establishing the positions of certain metals and alloys in a galvanic series.

The most convenient arrangement for study of the galvanic behavior of metals and alloys in flowing sea water was to use specimens of the materials listed in Table V in the form of pipe through which the sea water passed continuously. The design of the apparatus in current use was based on experience gained in the operation of a similar setup for about 600 days. The results of this first series of tests will not be discussed in detail, but will be used to supplement the data obtained from the present setup. In the previous case, sea water was allowed to flow through the test assembly throughout the year, while with the present setup the salt water was shut off, the specimens were washed with fresh water, and were then allowed to remain dry during the late spring, summer, and early autumn to avoid the attachment of marine organisms which might otherwise have covered the surfaces of the specimens and clogged the lines. As a result, the present results apply particularly to exposure during the period from December to April when there was no growth of marine organisms on the specimens.

Preparation and Arrangement of Specimens:

The specimens were in the form of standard 2-in. iron pipe size nipples 4 in. long. The exposed area inside was 26 sq. in. A nickel lug, measuring $\frac{1}{2}$ by $\frac{3}{4}$ by $\frac{1}{16}$ in. was silver brazed to each specimen as a means of attaching the wires to be used in making the current and potential measurements.

In order to develop a uniform surface condition, all the specimens except the galvanized wrought iron were pickled and scrubbed with pumice, both inside and out, after the nickel lugs had been attached by silver soldering. The galvanized specimens were cleaned by scrubbing with pumice after the nickel lugs had been attached by soft soldering. A similar procedure for attaching the nickel lugs was followed in the case of the lead specimens.

After the specimens had been cleaned as described, they were rinsed in alcohol and ether, dried, and weighed with an accuracy of ±0.01 g. They were then carefully wrapped in tissue paper and kept dry until they were assembled into the columns.

The specimens, in groups of ten, were assembled into columns, using bakelite impregnated fiber for insulating couplings between specimens as shown in Fig. 1. In addition, soft rubber gaskets were placed between the ends of specimens where they met inside the couplings. The joints between the rubber gaskets and the specimens and the threaded joints in the couplings were sealed with a cement containing a bakelite resin and zinc chromate. Joints made in this way were found to provide complete insulation between specimens and were completely watertight as well. The cemented gasket joint should also avoid concentration cell or contact corrosion of the ends of the specimens where they bear against the rubber gaskets. In the first test setup, some of the stainless steel specimens suffered

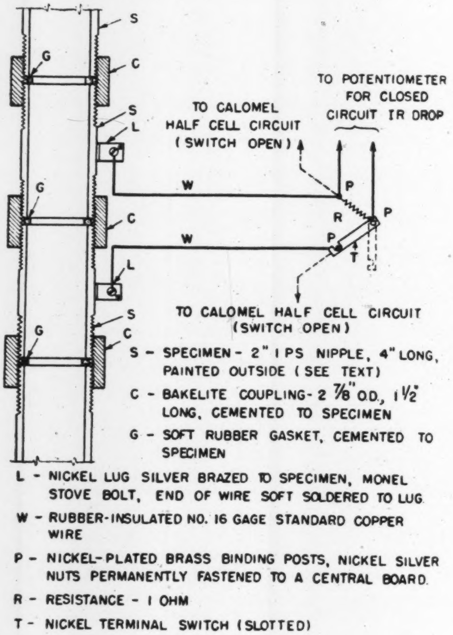
S
G
C
TO POTENTIOMETER FOR CLOSED CIRCUIT IR DROP
TO CALOMEL HALF CELL CIRCUIT (SWITCH OPEN)
S
L
G
W
P
R
P
C
W
P
T
L
TO CALOMEL HALF CELL CIRCUIT (SWITCH OPEN)
S
G
C
S - SPECIMEN - 2" I PS NIPPLE, 4" LONG, PAINTED OUTSIDE (SEE TEXT)
C - BAKELITE COUPLING - 2 7/8" O.D., 1 1/2" LONG, CEMENTED TO SPECIMEN
G - SOFT RUBBER GASKET, CEMENTED TO SPECIMEN
L - NICKEL LUG SILVER BRAZED TO SPECIMEN, MONEL STOVE BOLT, END OF WIRE SOFT SOLDERED TO LUG
W - RUBBER-INSULATED NO. 16 GAGE STANDARD COPPER WIRE
P - NICKEL-PLATED BRASS BINDING POSTS, NICKEL SILVER NUTS PERMANENTLY FASTENED TO A CENTRAL BOARD
R - RESISTANCE - 1 OHM
T - NICKEL TERMINAL SWITCH (SLOTTED)

Fig. 1.—Method of Assembling Tubular Specimens and Electrical Connections.

contact corrosion under the rubber gaskets.

The wires for the electrical connections were fastened to the nickel lugs by means of monel bolts, nuts, and washers. The mechanical joints made in this way were supplemented by allowing extensions of the wires to project from beneath the nuts and washers and by soldering these free ends of the wires to the corners of the nickel lugs. This insured good, low-resistance electrical connections in every case.

After each column of ten specimens had been assembled and the wires had been attached, the entire outside of each column unit was painted. The paint was applied to the specimens so that it covered the electrical connections on the nickel lugs and any exposed wire adjacent to the lug from which the insulation had been removed.

The procedure for painting was as follows:

1. One coat of quick drying zinc chromate primer conforming to U. S. Navy Specification P-27 was applied by brushing and allowed to dry for 1 hr.

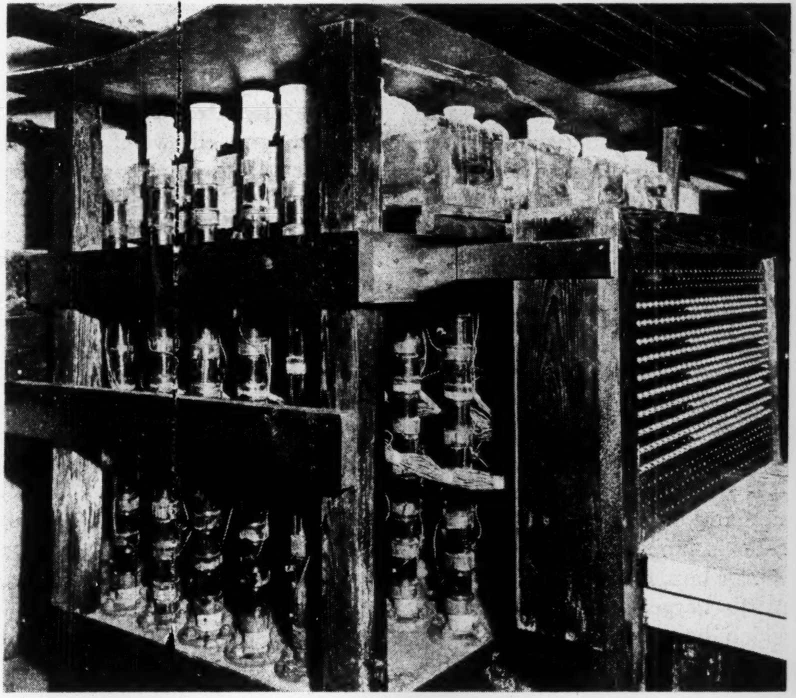

FIG. 2.—Pipe Test Assembly and Switchboard for Electrical Measurements.

2. One coat of a mixture containing one part of this primer and one part of a quick drying rubber base paint was applied by brushing and allowed to dry for at least 1 hr.

3. Two coats of rubber base paint were applied by brushing, with the first coat being allowed to dry overnight before applying the second.

This method of protecting the outside of the specimens was chosen on the basis of previous experience, which had shown it to furnish excellent protection in marine atmospheres and under conditions of exposure to condensed moisture and occasional wetting by sea water.

Thirty columns of specimens were prepared in the manner described. These were set up on top of a sea water supply manifold, or distributor, in five rows of six columns each as shown in Fig. 2. The connections from the columns to the manifold were made by means of copper-nickel alloy nipples and monel flanges fastened to a copper-nickel alloy cover plate. The nipples were assembled into the columns and insulated from the adjoining specimens in exactly the same manner as the specimens themselves, as described previously. A similar practice was followed in connection with the nipples at the tops of the columns through which the water was discharged through an assembly consisting of a tee, a nipple, and an elbow.

The original water supply manifold was a wooden box which was destroyed finally by teredo attack. The present manifold is made of steel and is painted both inside and out.

Sea water is supplied to the manifold, or distributor box, from the main, 72-in. diameter, discharge line of a centrifugal pump which has a capacity of 60,000 gal. per min., and which furnishes sea water to the plant of the Ethyl Dow Chemical Co. at Wilmington, N. C. The connection to this source of supply is made by means of 3-in. iron pipe size pipe through which the flow of water is controlled by means of a 3-in. globe valve and a 6-in. gate valve. The latter is placed between a 6-in. tee and the 72-in. pipe to provide a main shut-off point. A $\frac{1}{4}$-in. mesh monel screen is inserted in the 6-in. tee to prevent the passage of materials that might clog the test columns. The 6-in. valve is left completely open, and the rate of flow is controlled by means of the 3-in. valve.

The connection to the manifold, or distributor box, is made at the center of the bottom, and the water as it enters is made to pass through an impeller type distributor and then through a perforated baffle plate in order to smooth out the flow and distribute the water uniformly among the columns.

The initial adjustment of flow so as to provide the same velocity in each column was made by allowing only a very small amount of water to enter the system. The discharge elbows of all but the four corner columns were turned so that the discharge lips were at their maximum height. The flow was then divided equally among the four corner columns by rotating the elbows so as to raise or lower the level of the discharge lips. It was possible to make very accurate adjustments in this way, and finally to arrange so that water just dripped out of each of these four corner columns. The remaining columns were then adjusted to match the flow of the corner columns using a similar technique so that finally a condition was reached where water would drip slowly out of each column. When the flow of water was increased, all columns were found to discharge the same volume of water as indicated by the size of the stream flowing over the lip of each discharge elbow. It was estimated that the variation in flow among the columns did not exceed ±5 per cent of the average.

The water discharged from each row of six columns was collected in monel lined troughs, through which it flowed to a main discharge trough, and finally through a 6-in. overflow pipe back to the ocean.

Electrical Connections:

A wire was fastened to each specimen as described previously. These wires were connected to binding posts on a switchboard as indicated in Figs. 1 and 2.

Since it was desired to study several metals and alloys in galvanic contact, the columns were arranged so that the specimens forming each galvanic couple were adjacent to each other. The wires from these specimens were fastened to binding posts similarly located adjacent to each other on the switchboard. An additional binding post was furnished for each of these couples, and a calibrated 1-ohm resistance was placed between this third binding post and that connected to one of the specimens forming the couple as indicated in Fig. 1. The galvanic circuit was completed by means of a slotted nickel strip which was placed across the binding posts on the front of the panel and by means of which the galvanic circuit through the 1-ohm resistor could be opened or closed at will. All connections at the switchboard were soldered and covered for protection with petroleum jelly applied hot. The back of the switchboard was inclosed.

When a circuit was closed by means of the nickel switch, it was possible to measure the value of the galvanic current by determining the potential drop across the 1-ohm resistance as indicated in Fig. 1. This *IR* drop, as measured in millivolts by means of a potentiometer, could be recorded directly as milliamperes since the resistance was unity. The total resistance of the external circuit did not exceed 1.1 ohms.

This method of arranging the galvanic circuits made it possible to measure current flow at any time without disturbing any of the electrical connections. It was also possible to break the circuit at any time for open-circuit potential measurements.

In addition to the specimens forming the galvanic couples, which were set up in duplicate, at least two uncoupled specimens of each material were included in the assembly. The wires from these specimens were merely fastened to appropriate binding posts on the switchboard so that their potentials with respect to each other, or to a standard reference electrode, could be determined readily.

The standard reference electrode used in these studies was a saturated calomel half cell which was placed in the circuit by allowing the tip of the half cell to dip into the water in the overflow box.

TABLE VI.—TYPICAL ANALYSIS OF SEA WATER IN VICINITY OF WILMINGTON, N. C.

Constituent	Concentration, ppm.
Sodium as Na	10 590
Chlorine as Cl	19 200
Magnesium as Mg	1 292
Calcium as Ca	404
Potassium as K	403
Sulfate as SO_4	2 664
Phosphate as PO_4	less than 1
Boron as BO_3	10
Bromine as Br	67
Iron as Fe	negligible
Carbonate as CO_3	negligible
Total solids in solution	34 550
Total solids in suspension	5.7
Organic material in suspension	1.6
pH (glass electrode)	7.7

Conditions of Exposure:

Under normal conditions the total volume of water supplied to the system was 70 gal. per min. under a head of about 15 ft. The equivalent velocity through each column was about 15 ft. per min. Since the water contained some fine sand in suspension, the screen, valves, and distributor box gradually filled with sand, and the volume of water gradually decreased. However, the system was never allowed to remain stagnant, since at regular intervals the distributor box and connecting pipes were flushed out by back washing through connections provided for the purpose, and the screen was cleaned at the same time or more frequently as might be required.

The sea water was free from pollution, and was well aerated through the action of breakers at the entrance to the pump suction pond. A typical analysis is given in Table VI. The temperature of the water varied with the seasons between approximately 10 and 30 C. (50 and 86 F.). However, as indicated previously, the present tests were made only during the late fall, winter, and early spring when the range of temperature was between approximately 10 and 25 C. (50 and 77 F.).

In order to determine to what extent corrosion of the specimens and the accessory metal equipment might increase the metal content of the water used for testing, samples of water taken directly from the ocean and after having passed through the assembly were subjected to spectroscopic analysis by the Aluminum Research Laboratories through the courtesy of E. H. Dix. This study showed no detectable change in the metal content of the water.

Electrical Measurements:

Potentials were measured with a portable potentiometer with an accuracy of ±0.5 mv. for values over 10 mv., and ±0.05 mv. for values between 0.1 and 10 mv.

The following determinations were made:

1. Open-circuit potentials of all specimens relative to the calomel half cell at the start of the test and at various intervals throughout the exposure period.
2. The values of the current flow in the several galvanic circuits at the start of the test and at intervals throughout the test period. (This provided data on the changes in the value of current flow with time without breaking the galvanic circuit between successive current measurements.)
3. The temperature of the water, determined each time the electrical measurements were made.

In addition to these data, the weight losses of the specimens will be determined at the end of the test, and in the case of the galvanic couples, the galvanic effects as indicated by the electrical measurements will be correlated with the same effects as shown by the weight-loss data.

Results and Discussion:

Since these tests are still in progress, it is not possible at this time to present final results. However, the data obtained from the open circuit measurements during the first 173 days have been plotted in Figs. 3 and 4. In each case potential values less noble than the calomel half cell have been plotted above the zero line and have been indicated as anodic values. Similarly, values more noble than calomel have been plotted below the zero line and have been indicated as cathodic values. The anodic values, if written, should be preceded by a minus sign and the cathodic values with a plus sign.

During the test period, sea water flowed through the specimens continuously for 106 days from December 21 to April 6. The sea water was then shut off and the columns were flushed with fresh water and drained and left dry for an additional 65 days. Then the sea water was turned on again for two days in order to determine the potentials of the specimens in the warmer water at that time. The data plotted for the 173-day points in Figs. 3 and 4 were those observed on June 12, the second day after this shutdown period.

The variation in temperature throughout the test period is plotted in Figs.

3 (*c*) and 4 (*c*) from which it would appear that the general tendency for most of the materials to respond in the same way to changes in the water was affected to some extent by the temperature of the water, with most materials becoming less noble in the warmer water. Furthermore, the tendency for several materials to respond in the same way to changes in their environment indicated that while the potentials of each material varied through quite wide limits, the relative potentials remained fairly constant.

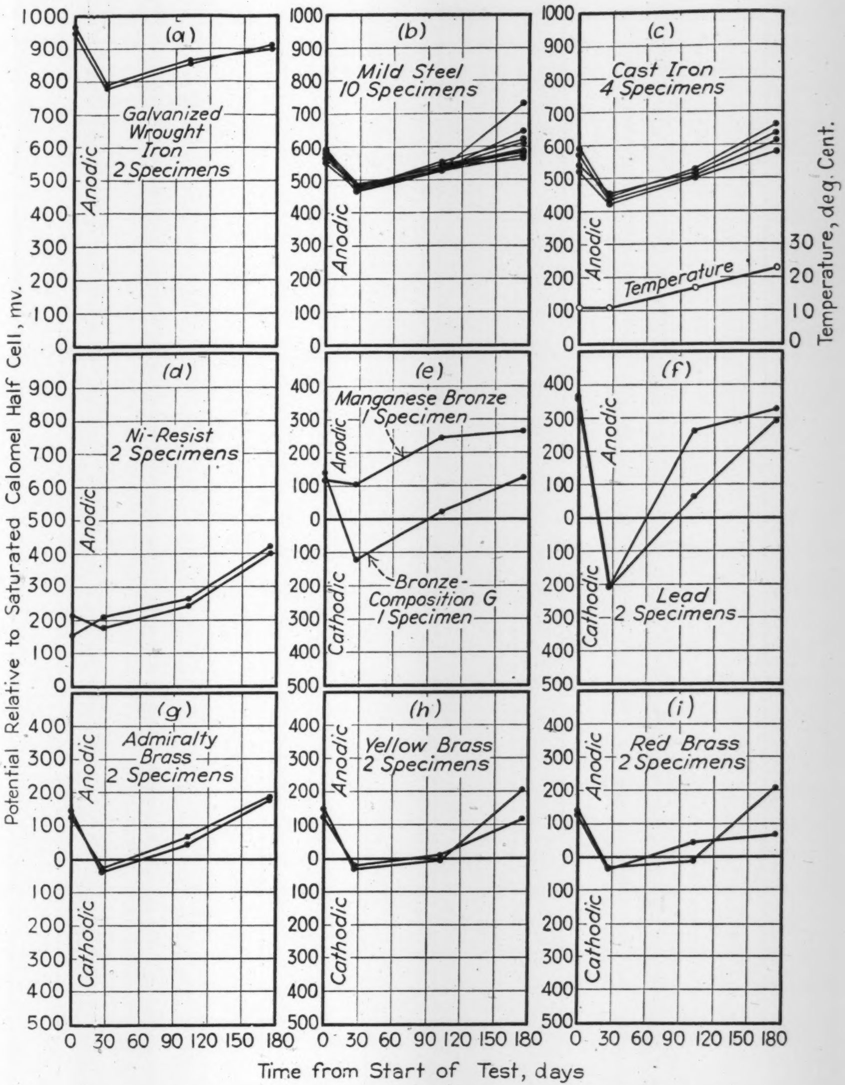

FIG. 3.—Potentials of Metals and Alloys in Flowing Sea Water.

In view of the controlling effect of surface films on the behavior of the stainless steels, it is not surprising that duplicate specimens of these materials should have shown a greater spread in values than was the case with the other materials. Even a small active area on an otherwise passive specimen would result immediately in a considerable rise in potential relative to the calomel half cell.

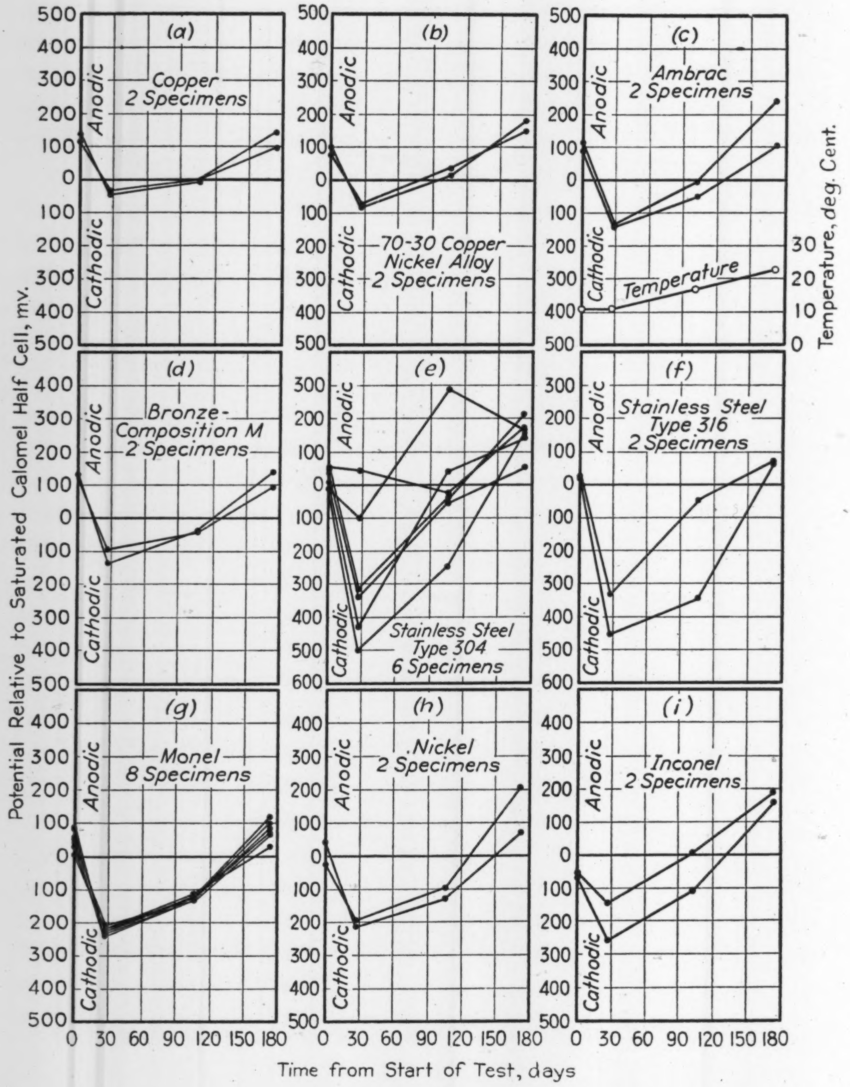

FIG. 4.—Potentials of Metals and Alloys in Flowing Sea Water.

The potential values for the types 304 and 316 stainless steel specimens at the 28-day points in the potential time curves, Fig. 4 (*e*) and (*f*), indicated a surprising degree of nobility. In fact, the maximum value for one of the type 304 specimens was 0.50 v. more noble than the saturated calomel half cell. This indicated a degree of nobility greater than that of platinum as determined by Uhlig (11) (see Table III). The authors have been unable to account for this. Observations of the potentials of other specimens made just before and just after the readings on these stainless steel specimens showed normal values. More recent observations have shown similarly high degrees of nobility for stainless steel specimens in the same test setup with the sea water at the same temperature as previously. In this latter case, special precautions were taken to insure that the calomel half cell used was functioning normally just prior to and just after the observations on the stainless steel specimens. Consequently, it must be concluded that these specimens actually were as noble as they appeared to be.

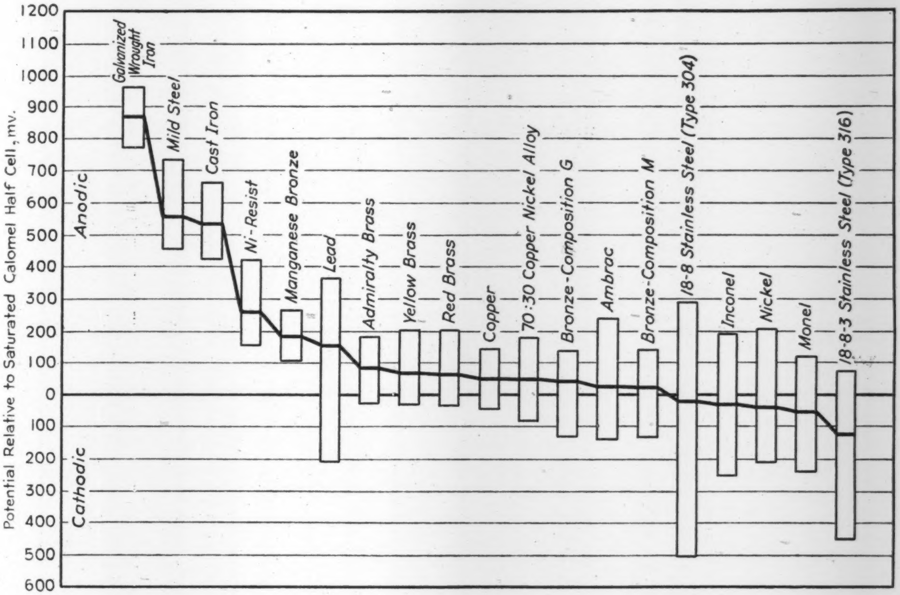

FIG. 5.—Galvanic Series Based on Ranges of Potentials of Metals and Alloys in Flowing Sea Water.

Apparently, also, the film that formed on lead in cold sea water made it relatively noble, although this nobility was not retained as the water became warmer.

In order to determine whether the position of a specimen in a column might affect its potential, one column was made up to contain six mild steel specimens distributed through the column, including the positions at the very top and very bottom. Similar practice was followed in the case of a series of eight monel specimens. The data referring to these specimens have been included in Figs. 3 (*b*) and 4 (*e*) from which it is evident that position in the column had no appreciable effect on the results obtained with either steel or monel.

An arrangement of all the materials in a galvanic series from left to right is shown in Fig. 5, with the maximum, minimum, and average values for each material being indicated. This method of presentation shows the probable direc-

TABLE VII.—GALVANIC SERIES FOR SEA WATER AS DETERMINED BY SEVERAL TESTS.

Series 1	Series 2 Figures are Potentials in Volts Referred to Saturated Calomel Half Cell		Series 3	Series 4
	Galvanized steel	−0.60		
Galvanized wrought iron	Galvanized wrought iron	−0.59	Galvanized wrought iron	Mild steel
Cast iron	Cast iron	−0.48	Mild steel	Ni-Resist
Mild steel	Mild steel	−0.45	Cast iron	50-50 lead tin solder
	Wrought iron	−0.45		
Ni-Resist	Ni-Resist	−0.39	Ni-Resist	18-8 stainless steel type 304 (active)
				Admiralty brass
18-8 stainless steel type 304 (active)	18-8 stainless steel type 304 (active)	−0.28	18-8 stainless steel type 304 (active)	Copper
Admiralty brass	Muntz metal	−0.27	Muntz metal	70-30 copper-nickel alloy
Red brass	Nickel (active)	−0.24	Yellow brass	18-8 stainless steel type 304 (passive)
Copper	Yellow brass	−0.20	Admiralty brass	Nickel (passive)
70-30 copper-nickel	Ambrac	−0.17	Red brass	Monel
Comp. M-bronze	Aluminum bronze	−0.17	Comp. M-bronze	18-8-3 stainless steel type 316 (passive)
18-8 stainless steel type 304 (passive)	Copper	−0.17	Copper	
Monel	Inconel (active)	−0.17	70-30 copper-nickel	
	Red brass	−0.15	18-8 stainless steel type 304 (passive)	
	Silicon bronze	−0.15	Monel	
	Admiralty brass	−0.14		
	Comp. M-bronze	−0.14		
	70-30 copper-nickel	−0.13		
	18-8 stainless steel type 304 (passive)	−0.08		
	Monel	−0.05		
	18-8-3 stainless steel type 316 (passive)	−0.04		

Series 5		Series 6		Series 7	Series 8
Continuous Immersion	Intermittent Immersion at Half Tide Level	Continuous Immersion	Intermittent Immersion at Half Tide Level	Sea Water Flowing Through Tubular Specimens	Sea Water Flowing Through Tubular Specimens
Mild steel	Mild steel	Cast iron	Cast iron	Galvanized wrought iron	Galvanized wrought iron
Cast iron	Cast iron	Mild steel	Mild steel	Mild steel	Mild steel
Ni-Resist	13% chromium stainless steel—type 410	Ni-Resist	Ni-Resist	Cast iron	Cast iron
13% chromium stainless steel—type 410	Ni-Resist	13% chromium stainless steel—type 410	13% chromium stainless steel—type 410	18-8 stainless steel type 304 (active)	Ni-Resist
Copper	18-8 stainless steel—type 304 (active)	Copper	70-30 copper-nickel	Ni-Resist	18-8 stainless steel type 304 (active)
70-30 copper-nickel	Copper	70-30 copper-nickel	Nickel	Lead	Manganese bronze
Nickel	70-30 copper-nickel	Nickel	Copper	Admiralty brass	Lead
Monel	Nickel	Monel	Monel	Copper	Admiralty brass
18-8 stainless steel—type 304	Monel	18-8 stainless steel—type 304	18-8 stainless steel—type 304	70-30 copper-nickel	Yellow brass
				Comp. M-bronze	Red brass
				Monel	Copper
				18-8 stainless steel—type 304 (passive)	70-30 copper-nickel
				18-8-3 stainless steel—type 316 (passive)	Comp. G-bronze
					Ambrac
					Comp. M-bronze
					Inconel
					Nickel
					Monel
					18-8 stainless steel type 304 (passive)
					18-8-3 stainless steel—type 316 (passive)

Bases of Several Galvanic Series in Table VII:

Series 1.—Based on directions of current flow in first 600-day test with pipe setup.

Series 2.—Based on average potential values relative to saturated calomel half cell in first 600-day test with pipe setup.

Series 3.—Based on direction of galvanic effects as calculated from weight loss determinations in first 600-day test with pipe setup.

Series 4.—Based on direction of galvanic effects as calculated from weight loss determinations on specimens immersed on spool type specimen holders in Barnegat Bay, N. J., for 124 days during summer and fall.

Series 5.—Based on direction of galvanic effects as calculated from weight loss determinations on specimens exposed on spool type specimen holders immersed continuously and intermittently in sea water at Eastport, Maine, for 274 days.

Series 6.—Based on direction of galvanic effects as calculated from weight loss determinations on specimens exposed on spool-type specimen holders immersed continuously and intermittently in sea water at Wilmington, N. C., for 180 days.

Series 7.—Based on directions of current flow in second 173-day test with pipe setup.

Series 8.—Based on average potential values relative to saturated calomel half cell in second 173-day test with pipe setup.

tions of galvanic effects, and also indicates which materials overlap so that variations in the direction of galvanic current flow may ocur. In connection with this latter point, however, it should be noted that in most cases changes in the potentials of the metals and alloys were similar in direction and extent, so that even though the potential of each material fluctuated, the potentials relative to each other remained fairly constant. The most important exceptions were in the cases of the stainless steels which may, therefore, exhibit erratic behavior with respect to their galvanic relationships toward each other and toward copper, nickel, and their alloys.

RESULTS OF OTHER INVESTIGATIONS BY THE AUTHORS

From time to time during the past few years, galvanic couples have been included in sea water corrosion tests carried out by the authors and their associates. Most of these tests have been made using the spool-type specimen holder and specimens which were described in an earlier paper by one of the authors (**18**). With these devices, the galvanic contact between specimens was made by using metallic spacers between the specimens. The extent and direction of the galvanic effects were determined by comparing the weight losses of the specimens in the couples with the weight losses of uncoupled specimens exposed in the same way at the same time. Galvanic series based on data of this sort are shown in Table VII which includes also a series based on the results of the 600-day test with the pipe setup that preceded the one described in this paper.

On the basis of all the data presented, including those taken from the literature, an electromotive, or galvanic series in sea water has been set up in Table VIII.

The metals and alloys have been divided into groups. Materials may change places within any group, depending on the incidental conditions of exposure. Likewise, galvanic effects in combinations of materials with others in the same group are not likely to be appreciable under most conditions. With any other combinations, the intensity of the galvanic effect will be determined by the relative areas of the materials forming the couple as discussed previously, by the distance apart in this series, and by the incidental conditions of exposure which affect corrosion reactions. In general, it is unwise to combine materials where the area of the material higher in the list is relatively small compared with that of the material lower in the list. The reverse relationship is not likely to be troublesome. For example, experience has shown that the combination of monel bolts in iron condensers in salt water service does not accelerate the corrosion of either the iron structure or the monel bolts.

Because of the generally good agreement among the results of the several investigations referred to in this paper, and because of the nature of galvanic corrosion phenomena, the authors of this paper suggest that the galvanic corrosion testing program referred to in the opening paragraphs of this paper be abandoned in its present form in favor of devoting the available time and money to the clearing up of any controversial points that may be brought out in the discussion of this paper, and to further studies of such phases of the subject as the effects of relative areas and of the distribution of galvanic effects around the points of contact of specimens forming galvanic couples.

Acknowledgments:

The authors wish to express their appreciation to the Ethyl Dow Chemical Co., Wilmington, N. C., for permission to set up the testing apparatus in their pumping station, and for the invaluable assistance rendered by C. M. Shigley, manager of the plant, and his staff. They wish also to thank W. A. Wesley and other associates in the Development and Research Division of The International Nickel Co., Inc., for their advice and help in carrying out the tests described in this paper.

Specimens used in this investigation were supplied through the courtesy of the following companies:

The American Brass Co.
The Walworth Co.
The Reading Iron Co.
The National Tube Co.
The Wm. Powell Co.
The Norfolk Navy Yard
The Babcock & Wilcox Tube Co.

TABLE VIII.—GALVANIC SERIES IN SEA WATER.

Magnesium
Magnesium alloys

Zinc
Galvanized steel or galvanized wrought iron

Aluminum 52SH
Aluminum 4S
Aluminum 3S
Aluminum 2S
Aluminum 53S-T
Alclad

Cadmium

Aluminum A17S-T
Aluminum 17S-T
Aluminum 24S-T

Mild steel
Wrought iron
Cast iron

Ni-Resist

13% chromium stainless steel type 410 (active)

50-50 lead tin solder

18-8 stainless steel type 304 (active)
18-8-3 stainless steel type 316 (active)

Lead
Tin

Muntz metal
Manganese bronze
Naval brass

Nickel (active)
Inconel (active)

Yellow brass
Admiralty brass
Aluminum bronze
Red brass
Copper
Silicon bronze
Ambrac
70-30 copper nickel
Comp. G-bronze
Comp. M-bronze

Nickel (passive)
Inconel (passive)

Monel

18-8 stainless steel type 304 (passive)
18-8-3 stainless steel type 316 (passive)

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DISCUSSION

Mr. A. W. Tracy[1] (*presented in written form*).—The authors of this paper are to be congratulated for making public their very extensive work on the potentials of metals and alloys in sea water. Those who have attended the inspections of the International Nickel Co.'s tests at Wilmington, N. C. can vouch for the extensive amount of work the authors have done to obtain these valuable results.

I should like to be permitted to add emphasis to the authors' statement that galvanic tests can only be semi-quantitative. Changes in the composition of the environment, relative areas of the metals combined, and the nature of the contact between two metals may give significantly different results in galvanic corrosion.

For instance, the galvanic action between two metals in mechanical contact such as reported in the paper may be entirely different from the same two metals in what might be termed metallurgical contact, such as a hot-dipped coating where an intermediate alloy layer may be formed. Merica[2] reported on the corrosion of scratched tinned copper in atmospheric exposure and in some experiments in dilute acids showed that copper was cathodic to the tin layer but slightly anodic to the copper-tin alloy layer.

It has been known that badly scratched tinned copper sometimes corrodes at the bare copper areas in rapidly moving sea water, so it was decided to find out if the corrosion of the copper was due to the rapid removal of copper ions or whether galvanic corrosion due to the alloy layer was responsible.

Two-inch lengths of copper pipe 0.311 in. inside diameter were tinned in a molten bath of new Banka tin. The tin and eutectic layer were removed from one sample by immersing in warm hydrochloric acid to expose the copper-tin alloy layer. A bare copper tube was connected by a rubber tube to a tinned copper tube as dipped and a bare copper tube was connected by a rubber tube to a copper tube with the alloy coating. Electrical connections were made by soldering copper wires to the outsides of the metal tubes to make galvanic contact and to make the potential measurements. The couples were connected to a pump which could circulate 3 per cent Turk Island sea salt solution at 32 ± 2C. through the tubes at 5 ft. per sec. Initial readings were made with the solution quiet. Each couple was short circuited through the copper wires except when potential measurements were being taken. The results of the tests are given below:

POTENTIAL OF COPPER *VERSUS* HOT-TIN COATINGS IN 3 PER CENT SEA SALT SOLUTION, MILLIVOLTS

	Copper (Cathode) *versus* Tin (Anode)	Copper (Anode) *versus* Alloy Layer (Cathode)
Initial Potentials, quiet solution	250	20
Initial Potentials, solution moving at 5 ft per sec.	150	40
2 hr. at 5 ft per sec.	150	70
24 hr. at 5 ft. per sec.	150	50
48 hr. at 5 ft. per sec.	92	77
48 hr., solution quiet for several minutes	198	20

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[1] Assistant Metallurgist, The American Brass Co., Waterbury, Conn.

[2] P. D Merica, "Structure of the Coating on Tinned Sheet Copper in Relation to a Specific Case of Corrosion," *Technical Paper No. 90*, Nat. Bureau Standards (1917); also *Transactions*, Am. Inst. Metals, Vol. 10, pp. 109-118 (1916).

Tests on a cell containing an electrolytic copper sheet electrode and a cast speculum metal electrode having a composition of 38.35 per cent tin and 61.65 per cent copper in a quiet 3 per cent sea salt solution gave potential measurements of the same order as the bare copper tube connected to the tinned tube with exposed Cu_3Sn alloy when both couples were in the quiet solution.

The tests indicate that motion of sea water does have an effect in making the potential of copper less noble but the main factor in causing corrosion of copper at scratches would appear to be the galvanic potential between the alloy layer and bare copper.

The results of galvanic tests in sea water cannot be used to predict the corrosion effects of hot tinned copper in other media. Although it is known that copper will be corroded galvanically where rain water flows from a high tin solder onto copper, the same type of specimen in a salt spray box will show that the solder is anodic to copper. Hot tinned copper has been used extensively and successfully for many purposes. Furthermore, much tinned copper sheet used today is electro-tinned and is free from the alloy layer.

MR. F. L. LAQUE.[3]—Inasmuch as Mr. Tracy's discussion was presented as a supplement to the data which we have presented, no detailed reply is indicated. However, I should like to take advantage of this opportunity to record some data which tend to confirm Mr. Tracy's observations. In the test setup described in this paper, there were troughs into which the sea water spilled from the pipe test columns. We used one of these troughs to conduct a test similar to that carried out by Mr. Tracy. This trough was lined with copper coated with a 50:50 lead-tin solder. The solder coating was scraped so as to expose a small spot of bare copper in the bottom of the trough. The location of this bared spot was recorded with reference to a mark placed on the side of the trough before the test was started. After sea water had spilled into the trough and had flowed over the spot of bare copper for about three months it was discovered that the trough lining had become perforated at this point. The thickness of the copper was about 0.078 in., indicating a rate of corrosion of the exposed copper in excess of $\frac{1}{4}$ in. penetration per year.

This test did not establish whether the high rate of corrosion of the copper was due simply to the effect of velocity, or whether it was influenced by galvanic effects between the bare copper and the surrounding solder covered with a corrosion product film. However, it did demonstrate in a practical way that if a break should occur in the solder coating on copper exposed to flowing sea water, accelerated corrosion of such exposed copper may be expected. The rapidity of the attack could be accounted for readily by the assumption that solder exposed to flowing sea water acquires a corrosion product film which is more noble than bare copper. A few simple potential measurements have tended to support this assumption.

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CONTROLLING FACTORS IN GALVANIC CORROSION*

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Synopsis

Data are presented which show the effects of variation of cathode area and galvanic circuit resistance upon the corrosion current in tests on iron coupled with copper in a neutral sodium chloride solution. Anode and cathode polarization curves are given for iron and copper, respectively. Currents estimated from these by application of Evans' graphic method are in good agreement with the observed galvanic currents. It is further found possible to express the results by a simple equation relating the current, the anode and cathode areas and the resistance.

The principle that the galvanic corrosion current is directly proportional to the cathode area is deduced to be valid at moderate ratios of cathode area to anode area in solutions with good conductivity but is shown to be inapplicable at high ratios where the retarding effects of resistance and anode polarization become important. Practical aspects of galvanic corrosion are discussed, including the influence of the nature of the cathode material and the selection of safe combinations of dissimilar metals.

A number of years ago a study was made of the effect of variation in resistance of the galvanic circuit upon the current generated by contact of dissimilar metals in neutral and acid electrolytes. Subcommittee VIII on Galvanic and Electrolytic Corrosion of the Society's Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys learned of this and requested that some of these unpublished data be presented in a paper for discussion as a companion to the paper by LaQue and Cox[2] on potentials of metals in sea water. In the early work it had been found that for a wide variety of metal couples the results could be expressed amazingly well by an empirical equation of the following nature:

$$V = I\left(R + \frac{K}{A_c}\right) \qquad \text{(1)}$$

where I is the galvanic current, R is the total unpolarized resistance of the circuit, A_c the cathode area, K a constant and V a constant in most cases equal to the open circuit electromotive force of the couple. The chief exceptions were those in which platinum was included as one of a pair of dissimilar metals. In addition to the resistance experiments galvanic corrosion tests were also completed with the iron-copper couple in 3 per cent sodium chloride solution in which the cathode area was varied over a wide range. Here, again, Eq. 1 was found to apply with values of the constants identical with those determined in the corresponding resistance experiment.

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* The presentation of this paper at the annual meeting was sponsored by Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.

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[2] See p. 670.

It was recognized that this equation involved Ohm's Law and in fact reduces to Ohm's Law at low values of cathode polarization where K is small or A_c is large. However, the significance of the polarization term was not well understood and it was not until the graphic method of Evans[3] for deriving corrosion currents from polarization curves was applied that the results of these galvanic corrosion experiments have been satisfactorily explained. This has required determination of the anode and cathode polarization curves for iron and copper, respectively, under the corrosive conditions maintained during the cathode area and resistance experiments.

The experiments with the iron-copper couple in aerated 3 per cent sodium chloride solution will be described in detail. It was felt that the interests of Committee B-3 would be better served by a thorough analysis of the behavior of this one metal pair in one environment than by an attempt to discuss a number of galvanic couples in various media in a necessarily more general way.

APPARATUS AND PROCEDURE

In the galvanic corrosion tests the temperature, degree of aeration of the electrolyte and the rate of motion of the electrodes were each controlled by use of the "circular path" corrosion apparatus.[4] This provides a yoke from which the specimens are suspended and which imparts to them a uniform motion in a vertical circular path of 5.1 cm. diameter at a speed of 4.7 m. per min. This type of motion is particularly advantageous in galvanic testing because a commutator or mercury cup is not needed in making electrical connections to the test specimens. Since no twist is involved the leads from the electrodes can be directly connected to a calibrated resistance attached to the yoke. The degree of aeration of the electrolyte was controlled at the saturation point by introducing filtered air through porous alundum thimbles which serve to break up the air stream into minute bubbles. A glass chimney attached over the aerator prevented direct impingement of bubbles upon the specimens. The temperature in all experiments was maintained at 30 ± 1 C.

The anodes were cylinders of commercially pure ingot iron[5] 2.5 cm. in diameter and 0.6 cm. thick with an 0.6-cm. hole drilled through the center. An enameled copper wire was soldered to the rim and the joint, rim and one face of the specimen were coated with paraffin. The cathodes were of commercial rolled sheet copper.[6] Each was paraffined on the back surface and an anode was mounted against the middle of the front face by means of an insulated rod passing through both anode and cathode. The paraffin on the anode and a 2.5-cm. disk of thin sheet rubber served to insulate the electrodes from each other. The anode surface was thus a disk situated about 0.9 cm. from the plane cathode surface and parallel to it. Just before starting each experiment the cathode was cleaned by dipping it in a bright pickling solution after which it was rinsed successively in pure concentrated sulfuric acid solution and water. Perfect freedom of the cathode surface from grease or oily matter seemed essential for reproducibility of results.

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[3] U. R. Evans, "Distribution and Velocity of the Corrosion of Metals," *Journal*, Franklin Inst., Vol. 208, No. 1, p. 52 (1929).

[4] O. B. J. Fraser, D. E. Ackerman and J. W. Sands, "Controllable Variables in the Quantitative Study of the Submerged Corrosion of Metals," *Industrial and Engineering Chemistry*, Vol. 19, p. 332 (1927).

[5] Nominal composition 99.8 + per cent iron.

[6] Nominal composition 99.9 + per cent copper.

The galvanic circuit was completed by an external resistance of 0.238 ohms, the potential across which was measured in determining the current by means of a potentiometer equipped with a sensitive galvanometer. In the cathode area experiments the duration of each test was about 18 hr. so that it was more convenient to measure the average current by determining the total loss in weight of the anode and calculating from it the equivalent average galvanic current. This is permissible when the anode area is so small that its normal rate of corrosion is negligible in comparison with the rate of galvanic corrosion, a point well established in preliminary tests as well as by published results of corrosion current experiments.[7] In the experiments in which the external resistance was varied the electrodes were connected through an external resistance of 0.238 ohms until a steady current was reached. The potential drop across this resistance was then recorded and a resistance of 2 ohms was inserted in series. After waiting 10 min. the current was again recorded and so on. The internal resistance of the galvanic cells was measured with 60-cycle alternating cur-

TABLE I.—EFFECT OF CATHODE AREA ON CORROSION OF IRON ANODES IN 3 PER CENT SODIUM CHLORIDE SOLUTION.

Temperature, 30 C. Solution saturated with air. Volume of solution, 60.6 liters.

Velocity of motion of electrodes, 4.7 m. per min. External resistance, 0.24 ohm. Anode area, 0.046 sq. dm. A_a.

Area Of Copper, A_c, sq. dm.	Total Resistance, R, ohms	Anode Loss in Weight, g.	Time, min.	Average Current, I, amp.		
				Observed (from Anode Loss)	Determined Graphically	Calculated from Eq. 10
1.01....	6.44	0.2267	1140	0.0115	0.0100	0.0115
2.97....	5.3[a]	0.5655	1085	0.0300	0.0241	0.0252
5.16....	4.3[a]	0.7920	1140	0.0400	0.0359	0.0366
8.35....	3.41	0.9405	1095	0.0495	0.0502	0.0505
10.00....	3.25[a]	......	....	0.0577[b]	0.0560	0.0555
11.63....	3.1[a]	1.0898	1095	0.0573	0.0605	0.0602
18.50....	2.94	1.2494	1080	0.0666	0.0720	0.0711

[a] Estimated.

[b] Observed steady state current.

TABLE II.—EFFECT OF RESISTANCE ON GALVANIC CORROSION.

Temperature, concentration, aeration, solution volume, and velocity of electrodes as in Table I.

Area of anode (iron) - 0.046 sq. dm. Area of cathode (copper)—8.40 sq. dm. Internal resistance—3.17 ohms.

Total Resistance, R, ohms	Current, I, amp.		
	Observed	Determined Graphically	Calculated from Eq. 10.
3.41......	0.0525	0.0505	0.0506
5.41......	0.0413	0.0392	0.0392
7.41......	0.0333	0.0320	0.0320
9.41......	0.0279	0.0271	0.0271
13.41......	0.0213	0.0208	0.0206
23.41......	0.0135	0.0134	0.0130
33.41......	0.0101	0.0099	0.0095

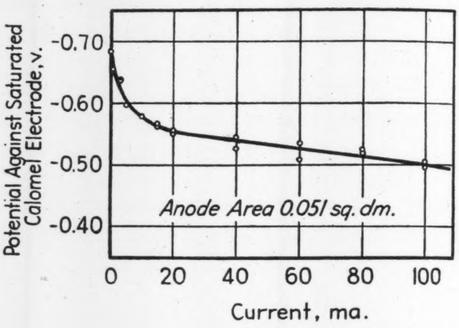

FIG. 1.—Anode Polarization of Iron in Aerated 3 per cent NaCl.

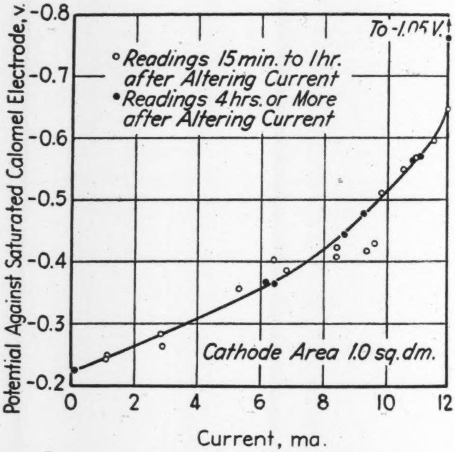

FIG. 2.—Cathode Polarization of Copper in Aerated 3 per cent NaCl.

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[7] W. A. Wesley, "Anode and Cathode Weight Losses in Galvanic Corrosion," *Transactions*, Electrochemical Soc., Vol. 73, p. 539 (1938).

rent with the aid of an alternating current galvanometer. This result was added to the external resistance to give the total resistance of the galvanic circuit, R.

The results of the galvanic corrosion experiments are presented in Tables I and II.

Polarization Curves:

A curve showing the variation of the potential of an anodic or cathodic surface with change in the current applied is called a polarization curve. For use in predicting the behavior of corrosion cells these relationships must be determined under corrosive conditions identical with those maintained in the cell; for example, the same temperature, degree of aeration and rate of motion of electrode or electrolyte must be continued. This was done in obtaining the anode and cathode curves for iron and copper, respectively, shown in Figs. 1 and 2. The potential in each case was measured with the aid of a tipped tube filled with the sodium chloride electrolyte, opening close against the electrode surface and leading through a sodium chloride - potassium chloride bridge to a saturated calomel electrode.

Little difficulty was experienced in obtaining steady state potentials for the anode polarization of iron but the cathode potentials of copper, particularly those at the higher current densities, were extremely sensitive to variation in the method of preparation of the cathode surface and to presence of any tarnish such as that which forms upon copper when very low cathode currents are applied. Most nearly reproducible results were obtained with copper cleaned by etching its surface in an ammoniacal solution of hydrogen peroxide followed by a scrub with a bristle brush dipped in clean pumice powder. It was observed that a steady state was reached more quickly when potential readings were made with successive increases in current rather than with decreases. The points in Fig. 2 were determined with successive increases in current. In addition, a few readings were made at indicated values of the current at which 4 to 18 hr. were allowed to pass without disturbing the circuit. Greater weight was given these steady state points in drawing the solid curve of Fig. 2.

EFFECT OF RESISTANCE OF GALVANIC CIRCUIT

Evans and Hoar[8] showed that the current in a corrosion cell can be computed from the electrode potentials by a modified form of Ohm's Law, whence

$$E_c - E_a = IR \qquad (2)$$

where E_a and E_c are the potentials of anode and cathode, respectively, as measured against a standard electrode. Since E_a and E_c vary with change in the corresponding current densities I/A_a and I/A_c it is necessary to determine the polarization curves before the corrosion current can be predicted over a range of values of R. It should be noted that R represents the total resistance of the galvanic circuit including the internal or electrolytic path as well as the external or metallic path of the current.

Polarization curves may be of complex shape because they often represent the effects of more than one electrode reaction in the region of potentials and current densities of interest in corrosion studies. In the case of the cathodic curve for copper in Fig. 2 the reduction of oxygen is probably the predominant reaction at potentials from -0.2 v. to the standard hydrogen electrode poten-

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[8] U. R. Evans and T. P. Hoar, "The Velocity of Corrosion from the Electrochemical Standpoint. Part II," *Proceedings*, Royal Soc. (London), A, Vol. 137, p. 343(1932).

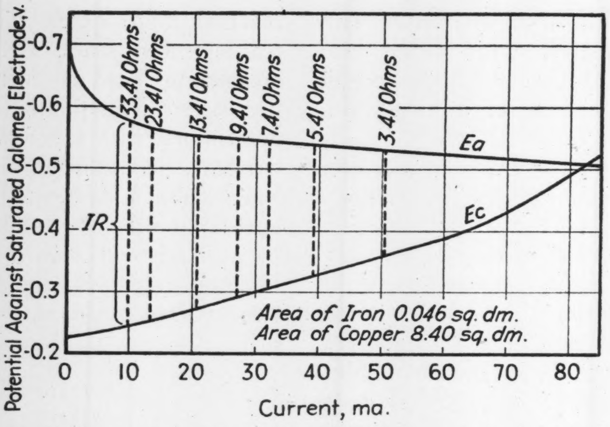

FIG. 3.—Graphic Determination of the Galvanic Current at Various Values of Resistance.

tial of the solution, namely, -0.66 v. with reference to the saturated calomel electrode. As the cathode potential is made increasingly negative there is a shift through an unstable region which should then lead to the usual logarithmic hydrogen overvoltage curve for copper beginning at potentials a few tenths of a volt more negative than -0.66 volt.[9]

When the polarization curves are of complex shape the derivation of the galvanic current from Eq. 2 can be best expressed graphically, following Evans.[3] To apply this method to derive the current-resistance relationship of Table II the iron and copper polarization curves are redrawn to correspond with the actual areas of iron and copper used in this experiment. This is done in Fig. 3. Then, for a given value of R, the current I which will satisfy Eq. 2 is that value at which the product IR equals the difference between the anode and cathode potentials, in other words that value of I at which the vertical distance between the two polarization curves is equal to the product IR. The dotted lines in Fig. 3 represent the values of I at which this is true for each value of R recorded in Table II.

The relationship between I and R thus derived graphically is shown as a curve in Fig. 4. The excellent agreement with the observed currents is demonstrated by plotting these as points on the same graph.

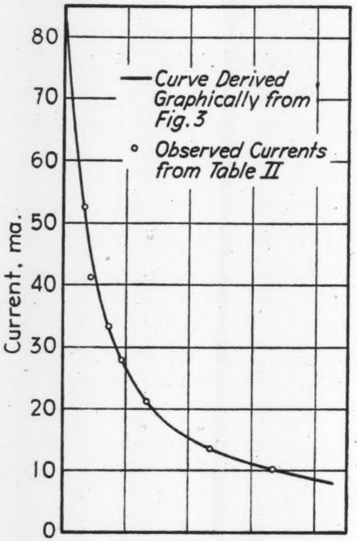

FIG. 4.—Effect of Resistance on Galvanic Corrosion.

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[9] O. Gatty and E. C. R. Spooner, "The Electrode Potential Behavior of Corroding Metals in Aqueous Solutions," p. 241, Oxford University Press, England (1938).

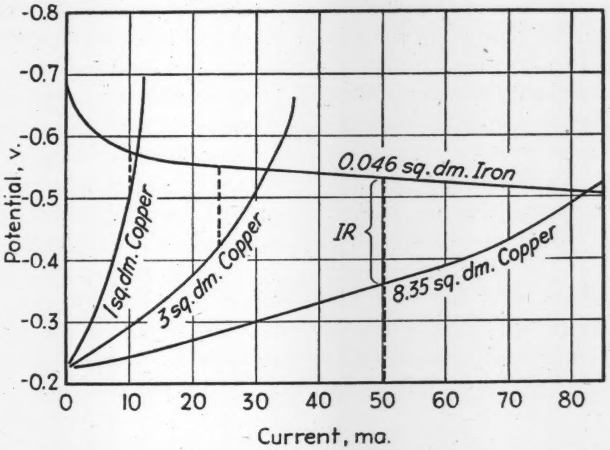

FIG. 5.—Graphic Determination of Galvanic Current with Different Cathode Areas.

There are two interesting deductions which can be made from Fig. 4. The first is that if it were possible to reduce R to zero the current would not become infinite but would reach the value 82.5 ma., the point at which the polarization curves intersect. This principle is important from an engineering standpoint because it establishes a maximum limit to the galvanic corrosion in a cell, useful if the resistance of the circuit were not known. The second is that there is no portion of the current-resistance curve at which I is independent of R. If the galvanic corrosion current were determined solely by the rate of diffusion of oxygen to the cathode surface, as is sometimes postulated, a variation in R would not affect I. The latter can occur only when the cathode polarization curve is vertical, as is true, for example, in the copper curve of Fig. 2 over a range of potentials only more negative than -0.65 v. Under the conditions of test maintained in the present experiments a liberal supply of oxygen is assured. If conditions were altered so as to restrict that supply then the current-resistance curve would be expected to show a range over which variation in R would not alter the value of I.

EFFECT OF VARIATION IN CATHODE AREA

Redrawing the original cathode polarization curves to represent the potential-current relationship for each area of

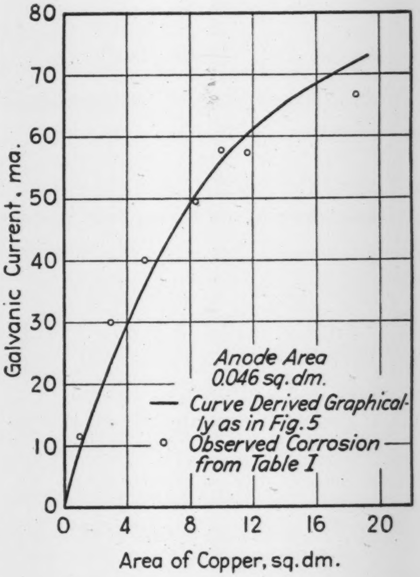

FIG. 6.—Effect of Cathode Area with High Ratio of A_c to A_a.

cathode employed in the experiments of Table I permits the ready graphic determination of the corresponding galvanic currents. If the resistance drop could be neglected the current would be given by the intersections of the anode curve with the corresponding cathode curves. Instead, correction for the resistance drop is made as before and is illustrated in Fig. 5. The results are presented in Table I and Fig. 6. The agreement between calculated and observed average currents is satisfactory considering the greater opportunities for error in the procedure used in determining the observed currents as compared with that employed in the resistance experiment.

The graphic method has been employed successfully by Brown and Mears[10] in deriving the effect of area of cathode upon the currents flowing between anodic and cathodic areas of aluminum, although the conditions employed were such that it was not necessary to determine values of R and the IR drop could be neglected.

"Catchment Area" Principle:

Whitman and Russell[11] exposed steel plates partly coated with copper to corrosion by water and found that, regardless of the fraction of surface coated, the total corrosion remained the same as that of the bare steel. They suggested that the velocity of attack was controlled by the amount of oxygen reaching the metal surface. The cathodic reaction took place over all the metal area and the corrosion was confined to the portion of steel not coated. Evans coined the term "Catchment Area Principle" for this theory, since the copper acted as a catchment area for oxygen. He has discussed this and other cathode area data[12] and pointed out that the principle should be applicable when the oxygen supply is indeed restricted enough that diffusion of oxygen is the sole controlling process, but that in general the rule is not quantitatively reliable. With a liberal supply of oxygen the principle could apply strictly only if the cathode polarization curves for the corroding metal and the more noble metal were identical.

The catchment area principle can be placed upon a truly quantitative basis by suitably modifying it. Consider the effects of variation in cathode area in cells in which the resistance drop IR and the differences in anode polarization are small enough to be neglected. The currents for different cathode areas are then given by the intersections of the individual cathode polarization curves with the substantially horizontal anode curve. Since the intersections occur at the same potential with all the cathodes they must represent the same cathode current *density.* The galvanic corrosion currents must then be directly proportional to the areas of copper connected to the iron. The reasoning will hold whether the electrolyte be neutral or acid, whether the cathode reaction be reduction of oxygen or liberation of hydrogen gas.

This quantitative principle can therefore be stated as follows: *When conditions are such that both anode polarization and resistance drop can be neglected the galvanic corrosion of the anode is directly proportional to the area of the cathode.* Call this the "constant current density" principle:

$$\text{Galvanic corrosion} = I = k\,A_c \quad (3)$$

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[10] R. H. Brown and R. B. Mears, "The Electrochemistry of Corrosion," *Transactions*, Electrochemical Soc., Vol. 74, p. 502 (1938).

[11] W. G. Whitman and R. P. Russell, "The Natural Water Corrosion of Steel in Contact with Copper," *Industrial and Engineering Chemistry*, Vol. 16, p. 276 (1924).

[12] U. R. Evans, "Metallic Corrosion, Passivity and Protection," pp. 513–516, Edward Arnold and Co., London (1938).

With negligible anode polarization the following relation would hold:

Total corrosion of anode = normal + galvanic corrosion =

$$k_1 A_a + k A_c \ldots\ldots\ldots (4)$$

Compare this with an expression of the catchment area principle:

Total corrosion of anode =

$$k_1 (A_a + A_c) \ldots\ldots\ldots (5)$$

It will be seen that Eq. 5 is valid only when $k_1 = k$. The catchment area principle is therefore expected to be reliable only when the anodic and cathodic materials of the couple are equally effective seats for the cathodic reaction, that is, when they have identical cathode polarization curves in the region of potentials close to the intersection with the anode curve. This statement is, of course, subject to the original assumption that the depressive effects of resistance and anode polarization must be negligible.

TABLE III.—ESTIMATED GALVANIC CORROSION FOR LOW RATIOS OF COPPER TO IRON AREAS.

Assume corrosive conditions same as those of Table I. The normal rate of corrosion of iron (alone) under these conditions was found experimentally to be 350 mg. per sq. dm. per day which is equivalent to 14 ma. per sq. dm.

Anode area in calculations below assumed to be 1 sq. dm.

Assumed Cathode Area, sq. dm.	Galvanic Current Estimated from Polarization Curves (R=0), ma.	Total Current (Galvanic plus Normal Corrosion), ma.	Total Current Predicted by "Catchment Area Principle," ma.
0........	....	14	14
1........	12.0	26	28
2........	23.9	38	42
3........	35.7	50	56
4........	47.5	61.5	70
5........	59.0	73	84

The situation can be clarified by discussion of specific couples. Both the constant current density principle and the catchment area principle are inapplicable in the data of Table I and Fig. 6 because the effects of R and of anode polarization are pronounced and cause the cathode area-current relationship to deviate widely from a straight line. The ratios of cathode area to anode area are very high and the quantitative relationships can be derived only graphically or by a formula to be discussed later.

Low Ratios of Cathode Area to Anode Area:

With low ratios of cathode to anode areas the depressive effects of resistance and anode polarization should be less pronounced. Let the corrosion which would take place in iron-copper couples in which the cathode to anode area ratio varies from zero to five be estimated. This can be done graphically with some degree of confidence by replotting the appropriate portions of the polarization curves of Figs. 1 and 2. Assuming that the anode area is relatively large (1.0 sq. dm.) and that the anode to cathode distance is not very great, the resistance of the circuit with 3 per cent sodium chloride electrolyte would be small

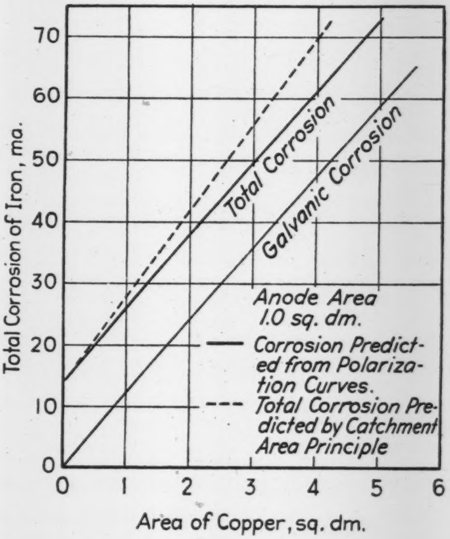

FIG. 7.—Effect of Cathode Area with Low Ratio of A_c to A_a.

enough to neglect. The galvanic corrosion to be expected is given in Table III. The total corrosion of the anode can be taken as approximately equal to the sum of the galvanic and normal rates in the case of iron corroding at moderate rates in neutral sodium chloride solution.[7]

The calculated corrosion results are illustrated in Fig. 7. Since they form a practically straight line it is evident that the constant current density principle (Eq. 3) is valid in this case. In addition, the catchment area principle appears to be not far from right for low ratios of copper to iron areas.

TABLE IV.— RATE OF CORROSION OF IRON COUPLED WITH VARIOUS CATHODES IN 3 PER CENT SODIUM CHLORIDE SOLUTION.

Corrosive conditions same as in Table I.

Area of anode - 0.14 sq. dm. Area of cathodes—3.3 sq. dm. Duration of test—6 hr.

Cathode Material (Freshly etched surface)	Loss in Weight of Anode, mg. per sq. dm. per day
Copper	9630[a]
Bronze	9670[a]
Brass	9430
Monel	8460

[a] Average of two experiments.

Effect of Nature of Cathode Metal:

The catchment area principle postulates that the nature of the cathode metal shall make no difference in the rate of galvanic corrosion. As explained before this would require that cathode polarization curves for different metals coincide. This would clearly be the case when the rate of the cathode reaction is limited solely by diffusion of oxygen. On the other hand, when the cathode reaction is chiefly the formation of hydrogen gas it is equally obvious that the cathode polarization curves for metals will differ in the same way as their hydrogen overvoltages differ. Evans[12] refers to a number of examples in the literature in which the rate of corrosion of iron or other anodes was accelerated equally by contact with different metals irrespective of their degree of nobility toward the anode. Some experiments performed in this laboratory agreeing with this are shown in Table IV.

Against these data Evans[12] placed some of his own. He tested strips of iron exposed to salt solutions (*a*) alone, (*b*) in galvanic contact with a strip of another metal of equal area, and (*c*) in galvanic contact with a strip of double area. The secondary metals were nickel, copper and lead. Evans pointed out that if the catchment area principle were valid the ratio of corrosion of iron in condition (*a*), (*b*), and (*c*) should be the same for the three metals and should be 1:2:3 in all cases. Actually, he found the ratio to hold well for nickel (1:1.91:2.86), but to be too low for lead (1:1.24:1.47), and too high for copper (1:2.80:4.17). It is interesting to point out now that Evans' experiments are in better agreement with the constant current density principle (Eq. 3). This is shown by subtracting the normal corrosion from his results in each case. Then the *galvanic* corrosion rates become for nickel (0:0.91:1.86), for lead (0:0.24:0.47), and for copper (0:1.80:3.17). In each case the results are not too far from being directly proportional to the cathode areas.

To recapitulate, the catchment area principle is a useful qualitative rule while the constant cathode current density principle is a true quantitative guide whenever the resistance drop in the galvanic cell and the change in anode polarization are small enough to be neglected.

EQUATION FOR GALVANIC CORROSION

In spite of the great progress which has been made in the past 10 yr. in understanding the mechanisms of electrode reactions involved in corrosion[13] it is still not possible to derive the cathode polarization curves for different metals from fundamental data. Fortunately, galvanic cells of practical interest often involve only relatively narrow ranges of current density so that the anode and cathode polarization curves can then be considered straight lines within such limits. This was true, for example, in the experiments of Evans and Hoar[8] with iron, and of Brown and Mears[10] with aluminum, in potassium chloride solutions.

If the polarization curves are straight lines they can be expressed by Eqs. 6 and 7:

$$E_a = E_a^0 + \frac{M_a I}{A_a} \ldots\ldots (6)$$

$$E_c = E_c^0 + \frac{M_c I}{A_c} \ldots\ldots (7)$$

where E_a^0 and E_c^0 are the intersections of the lines with the zero current axis (in most cases the same as the open-circuit potentials of the anodic and cathodic materials, respectively) and M_a and M_c are the slopes of the anodic and cathodic curves. Applying the fundamental equation of galvanic corrosion, namely

$$E_c - E_a = IR \ldots\ldots (2)$$

then

$$E_c^0 - E_a^0 = I\left(R + \frac{M_a}{A_a} - \frac{M_c}{A_c}\right). (8)$$

In many cases the difference $E_c^0 - E_a^0$ is the same as the open-circuit emf. of the galvanic couple concerned. Calling this difference V then

$$V = I\left(R + \frac{M_a}{A_a} - \frac{M_c}{A_c}\right) \ldots (9)$$

Equation 9 states the relationship between the galvanic current I and the resistance R and at the same time that between I and the cathode area A_c. It includes no arbitrary constants.

It is significant that this equation for galvanic corrosion reduces to the constant current density relationship (Eq. 3) when the terms R and M_a/A_a are small. Again, when the two polarization terms are small in comparison with R the equation reduces to Ohm's Law, $V = IR$, the significance of which in galvanic corrosion will be discussed later.

Equation 9 can be applied to the data of the resistance and cathode area experiments already presented. The iron polarization curve of Fig. 1 is a straight line for values of I greater than 20 ma. with the following constants:

$$E_a^0 = -\ 0.567 \text{ v. and } \frac{M_a}{A_a} = 0.71 \text{ if } A_a$$

is expressed in square decimeters. Similarly the copper polarization curve of Fig. 2 can be represented satisfactorily by a straight line over a range of current densities from zero to 9 ma. per sq. dm. by Eq. 7 with the following values of the constants:

$$E_c^0 = -0.217 \text{ v. and } \frac{M_c}{A_c} = -23.5.$$

Substituting these values of the constants in Eq. 9 gives

$$0.350 = I\left(R + 0.71 + \frac{23.5}{A_c}\right) .. (10)$$

The agreement between values of I calculated from Eq. 10 and the observed values for both the cathode area and the resistance experiments is shown in Tables I and II and is quite satisfactory.

If the equation were to be used to calculate the galvanic currents at low ratios of cathode to anode areas different constants would be used since the anode polarization would be low, making E_a^0 approach -0.68 v., and the cathode polarization would be high, where the cathode curve has a steeper slope.

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[13] Gatty and Spooner, *Loc. cit.*, Chapter I and Appendix.

It is interesting to compare Eq. 9 with the old empirical Eq. 1. They differ only in that the term which accounts for anode polarization (M_a/A_a) is missing from the latter. This deficiency was not important in applying Eq. 1 to the data on the iron-copper couple because the slope of the polarization curve for iron in sodium chloride solution is small. It is intended to review at a later date the early experiments which involved a variety of metal pairs exposed in acid as well as neutral solutions and apply polarization data now available which may be applicable to the specific combinations involved.

LIMITING OR MAXIMUM GALVANIC CORROSION

La Que and Cox[2] raise an important question in presenting their data on the potentials of metals and alloys in sea water. How far apart in their galvanic series can two metals be selected yet represent a couple safe to use in practice? There is one approach to the answer which may be of use to an engineer faced with a specific application in sea water, provided that the resistance of the galvanic circuit between the proposed parts can be estimated or measured:

The maximum value of the potential term $E_c - E_a$ in the fundamental Eq. 2 is that corresponding to the open circuit potential of the couple. This is true because polarization at either or both electrodes operates to reduce the difference $E_c - E_a$ if no external source of electromotive force is introduced. Calling the open-circuit potential difference V_o, measured under the corrosive conditions in question, then the maximum possible galvanic current is given by the relation

$$V_o = IR \text{ . . . . } (11)$$

It is therefore only necessary to substitute the emf. V_o estimated from the La Que and Cox series in Eq. 11 to obtain the value of the limiting galvanic current for the proposed cell whose resistance is R. If this current, converted to the equivalent rate of corrosion upon the anodic member of the galvanic couple and added to the rate of normal corrosion of the latter, proves to be a safe value then the selected pair of metals can be safely employed. If this limiting value is too high then an answer can be had only by determination of the polarization curves and application of Eq. 2.

ESTIMATION OF RESISTANCE R

It must be now evident that one of the important factors in galvanic corrosion is the resistance of the circuit R. It will be seldom possible to measure R exactly in studying galvanic corrosion problems in industry. However, even a crude approximation of the value of R could be useful from an engineering viewpoint. The resistance of the metallic portion of the galvanic circuit is usually negligible so that the problem resolves into determination of the resistance of the electrolytic portion of the circuit. This subject is dealt with by Jeans[14] and those who are concerned with galvanic corrosion problems will find this reference quite helpful. He discusses methods of mathematically approximating values of R for various shapes and sizes of electrodes. Of course the resistance of certain simple combinations like two concentric tubes can readily be computed. In other combinations it may be possible to fix a minimum limit for R even if the actual value cannot be estimated. This would then permit estimating a maximum value for the galvanic current.

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[14] J. H. Jeans, "The Mathematical Theory of Electricity and Magnetism," pp. 350-363, Cambridge University Press, England (1925).

PRACTICAL ASPECTS OF GALVANIC CORROSION

As a substitute for a set of specific conclusions it may be more useful to review galvanic theory from a practical and qualitative point of view. If a proposed application of dissimilar metals in contact is being considered and it is possible to obtain anode and cathode polarization curves and the resistance of the galvanic system then the galvanic corrosion can be estimated by the graphic methods already described. However, this information is not ordinarily readily obtainable and reliance must be placed upon general qualitative principles to avoid dangerous metal combinations.

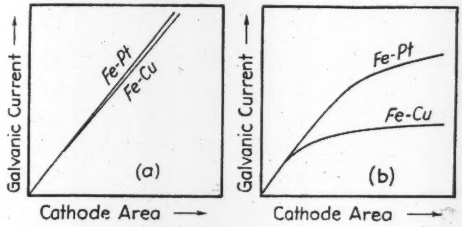

FIG. 8.—Effect of Relative Nobility of Cathode Material (Schematic).

(*a*) In salt solution with high conductivity such as sea water.

(*b*) In solution with poor conductivity such as tap water.

The current in galvanic corrosion increases with increase in area of the cathodic member of the couple. The maximum rate of increase is set by the constant current density principle which states that the galvanic current is directly proportional to the cathode area. In general this rule applies when the electrolyte is one of moderate or high conductivity, the anode does not polarize greatly and the cathode area is not too many times greater than the anode area. These conditions are commonly met by galvanic couples of many commercial metals in chloride solutions such as sea water. The relationship is illustrated by the lower solid curve of Fig. 7. The rule holds because the controlling factor under such conditions is the rate of the cathode reaction.

If it is known that the cathode reaction rate is itself limited by the rate of diffusion of oxygen, as in the corrosion of iron in sea water with a restricted supply of oxygen, then the nature of the cathode material is relatively unimportant, provided it is appreciably more noble than the anode material (Fig. 8 (*a*)). Furthermore, the total corrosion rate of the anode is here expected to vary directly as the total area of the couple, as illustrated by the upper curve of Fig. 7. These principles explain why it is safe to employ copper rivets on a steel plate exposed to sea water but not safe to use steel nails to fasten copper sheathing on a boat hull.

Where corrosion proceeds chiefly by liberation of hydrogen gas and the solution is a good conductor of electricity, the constant current density principle still should apply but the nature of the cathode metal will now play an important part. Under these conditions a cathode material of low hydrogen overvoltage will permit greater galvanic corrosion than one of higher overvoltage. Thus it is much safer to place zinc in contact with mercury in an acidic solution than with platinum.

At the other extreme in regard to controlling factors is the galvanic circuit in which the electrolyte resistance is high in relation to the available electromotive force of the metal pair. Here the cathode area, diffusion of oxygen and hydrogen over-voltage play minor roles because the galvanic current is determined solely by Ohm's Law, $V_o = IR$ where V_o is the open-circuit potential difference of the couple. The resistance and the available potential are the controlling factors. Thus, in very dilute solutions, iron will be expected to corrode more rapidly in contact with platinum than when in contact with an equal area of copper (Fig. 8 (*b*)).

The third class of galvanic conditions is the large one between the two extremes just discussed. Control does not shift suddenly from the cathode reaction type to the resistance type but there is instead a wide range of conditions over which the resistance of the circuit, the anode polarization and the cathode reaction all exert an influence upon the galvanic current. The data illustrated in Figs. 3 to 6 are in this class.

The foregoing generalizations can be rendered invalid by many disturbing factors, such as pronounced anode polarization, formation of protective films, nonuniform current distribution and the like. When these interferences occur it is necessary to determine anode and cathode polarization curves under the specific conditions involved in order to derive the galvanic corrosion currents.

Acknowledgment:

The guidance and encouragement of P. D. Merica, Vice-President of The International Nickel Co., Inc., are gratefully acknowledged, as are the many contributions of O. B. J. Fraser, now Director of Technical Service, under whose direction the galvanic corrosion tests were originally performed. The assistance of R. E. Smith, who determined the polarization curves, is greatly appreciated.

DISCUSSION

Mr. R. F. Passano.[1]—If the polarization curve for copper in Fig. 3 of Mr. Wesley's paper were extended beyond 90 ma., the potential of the copper would be more negative than that of the iron. Such a relation of potentials might be taken to mean that the copper was anodic to the iron. This position of the two metals is unnatural and cannot arise from galvanic currents. In spite of the "reversed" potential relations of the two metals beyond the intersection of the polarization curves, the copper is still acting as cathode and the iron as anode. The only way the copper could become an anode to the iron would be to hitch into the circuit, say, a dry cell battery and connect the carbon pole of the battery to the copper and the zinc pole of the battery to the iron. In this instance the electromotive force of the dry cell would buck the electromotive force of the galvanic copper-iron cell. The polarization curves for this artificially reversed condition would be very different from those the author has shown; there would be then an anodic polarization curve for the copper instead of the cathodic one shown. Is this substantially correct?

Mr. J. T. MacKenzie.[2]—We recently completed a test in artificial sea water for various combinations of copper-bearing and copper-free nuts and bolts which show that, while the copper-free part of a couple is attacked more than the copper bearing, the total corrosion in such an assembly with some copper in it is less than the total corrosion of a joint which has all copper-free materials in it. We made a test to see how much damage would be done if some mistake were made and we got copper-free nuts connected with copper-bearing bolts, etc. We were very pleasantly surprised to find that we had nothing to fear from such an assembly.

21-DAY TEST

Bolts				Nuts			
Joint	Type Iron	Number	Loss in Weight, per cent	Joint	Type Iron	Number	Loss in Weight, per cent
No. 1..	Plain	4	1.2	No. 1..	Plain	4	2.1
No. 2..	Cu—1%	4	0.86	No. 2..	Cu—1%	4	1.1
No. 3..	Plain	2	1.3	No. 3..	Plain	2	1.5
	Cu—1%	2	0.92		Cu—1%	2	1.2
No. 4..	Cu—1%	2	0.83	No. 4..	Plain	2	1.4
	Plain	2	0.97		Cu—1%	2	0.94

Another point in Mr. Wesley's tests is that the proportion of copper to iron is large, whereas in bronze welded assemblies, our main interest in cast iron pipe work, the proportion of iron to copper is very large; the corrosion depends on the access of oxygen to the small surface of copper, which means that it is inconsequential. In fact, we have a good many years' experience with bronze welded assemblies in sea water and there is very little localized corrosion around the weld. This has also been borne out in the Bureau of Standards Soil Corrosion Tests.

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[1] Research Engineer, The American Rolling Mill Co., Middletown, Ohio.

[2] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

MR. F. L. LAQUE.[3]—We have some data which parallel Mr. MacKenzie's observations on the behavior of brazed joints on cast iron exposed to corrosion. We have records of the use of monel bolts to join cast iron condenser sections submerged in salt water for over 20 yr. The cast iron in the vicinity of such bolts did not show any evidence of accelerated attack and, of course, the monel bolts were not affected in any way.

With reference to Mr. MacKenzie's experiences with cast irons containing a small amount of more noble alloying elements when used in conjunction with unalloyed cast iron, we have made some similar studies of the behavior of combinations of carbon steel and alloy steels. For example, nickel-steel rivets containing from 1.5 to 2 per cent nickel were driven into carbon steel plates and immersed in sea water. These alloy-steel rivets have remained substantially free from corrosion after 5-yr. exposure, whereas carbon steel rivets in the same plates have suffered considerable attack.

If the area relationships between an alloy steel or cast iron and unalloyed steel or iron are favorable, the small difference in potential which is developed may be sufficient to insure practically complete galvanic protection of the alloy without any significant increase in the corrosion of the unalloyed material.

MR. W. A. WESLEY[4] (*Author's closure*).—While the designer of metal equipment must guard against assembling dangerous galvanic couples, he should not overlook the possibility of putting galvanic effects to good use. Mr. MacKenzie and Mr. LaQue brought out interesting practical applications of beneficial galvanic corrosion. Note that in all three cases the ratio of cathode area (A_c) to anode area (A_a) was small and safe.

Mr. Passano pointed out a possible source of confusion in presenting graphs such as Fig. 3 and 5 due to the continuation of the polarization curves beyond their intersection. This disadvantage is mitigated by the fact that full curves do show more clearly how the graphs are constructed. His analysis of conditions required to reverse the direction of current in a given cell was instructive and correct. The reason why the potentials in a simple galvanic couple cannot move beyond the intersection of the polarization curves and thus reverse their relationship is evident from inspection of Eq. 2:

$$E_c - E_a = IR$$

As R is reduced the current I increases and E_c approaches E_a. E_c can become equal to E_a when R is made zero but it could become *less* than E_a only if R could become negative. Since R cannot be negative the reversed potential relations cannot occur. If an external source of electromotive force is inserted in the galvanic circuit, this introduces a third term into the left side of Eq. 2 and allows E_c to be forced to a more negative value than E_a. An external source of current was of course employed in obtaining the data for the polarization curves.

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[3] Research and Development Division, The International Nickel Co., Inc., New York, N. Y.

[4] Assistant Director, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

A CORROSION-FATIGUE TEST TO DETERMINE THE PROTECTIVE QUALITIES OF METALLIC PLATINGS

By John N. Kenyon[1]

Synopsis

This paper describes a corrosion-fatigue test for determining the extent of protection afforded by metallic platings. Steel bead wire (automobile tire reinforcement) was used for the investigation. It is believed that the results have general value in connection with fatigue phenomena and the various factors influencing fatigue failure.

It was shown that the measurements obtained by static corrosion and static physical tests, hitherto relied upon to determine the probable service qualities of bead wire, are inadequate for classifying these materials. Only fatigue tests wherein there is simultaneous corrosive attack, are sufficiently discriminatory for determining relative service qualities.

It was found that plating with a more noble metal, such as copper, may actually contribute to the embrittlement of steel wire by reason of numerous fatigue cracks resulting from galvanic action at points of imperfection in the plating. No embrittling conditions were encountered where the wire had first been galvanized before plating with copper.

Under the most favorable of service conditions, there is a slight deterioration in metals due to the gradual inroad of corrosion. These attacks may be so mild in nature that there is no appreciable loss in tensile and ductile properties, and only a slight loss in metal expressible in grams per square centimeters per year. If, on the other hand, the same metals are simultaneously subjected to cyclical stress, a corrosive attack, however slight, may have a pronounced effect on the endurance properties. Since slight corrosive attacks are considerably retarded by metallic platings, the present investigation was undertaken to determine the effect of some of these platings on the corrosion-fatigue resisting properties of steel wire.

Materials:

The material selected for the investigation is commercially termed "bead wire" used for automobile tire reinforcement.[2] It is subjected to cyclical stresses of a comparatively high order under widely varying conditions of environment. Inflational pressure in the tire exerts a steady tensile stress on the wire while centrifugal force, which varies with speed, exerts a more or less pulsating tensile stress and introduces the problem of fatigue. The wire also resists certain other forces of a vibrational and flexural nature due to uneven road beds and the rounding of curves; these, in general, may be classified as pulsating tensile stresses. In the early days of the automobile, bead wire failures were common. They are comparatively rare today due to improved methods of tire manufacture. Failures that do occur can probably be attributed to some form of fatigue fracture, see Fig. 1.

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[1] Instructor in Civil Engineering, Columbia University, New York, N. Y.

[2] H. C. Pearson, "Pneumatic Tires," p. 315, India Rubber Publishing Co. (1922).

Present-day bead wire construction (Figs. 2 and 3) is governed by the belief that the wire should be coated for protection and that it must adhere to the rubber. Adherence to vulcanized rubber is obtained by using copper-plated steel wire wherein the copper coating has in solution a small percentage of some other metal, such as tin, or zinc, or allied elements. The term "bronze plate" used hereafter refers to a copper coating with a trace of tin. The wire and rubber compound adhere to each other during vulcanization. While other metals might be used to give adherence to rubber compound, copper has been found by repeated tests to be the most practical plating material.[3,4]

Preliminary Investigation:

As an approach to the study of bead wire, a series of corrosion tests were made by a standardized method. This procedure was governed by the consideration that the automobile tire is exposed to all manner of road and weather conditions, extremes in temperature, and to highway contaminations. While the wire reinforcement is well vulcanized in rubber, it seemed reasonable to assume that this protection, due to injury or wear, may not necessarily be permanent. It also seemed reasonable to assume that the chemical components of the rubber might react chemically with moisture, or with extraneous materials, and attack the wire. The first step, therefore, was to consider some of these highway contaminating materials and their possible chemical effects.

The intermittent immersion test method was selected for the preliminary study, since the effect of chemical attacks are expressible in numerical terms. This test method was developed for studying the behaviors of metals when subjected to alternate wet and dry conditions; a diagram of the apparatus is given in a paper by Fink and

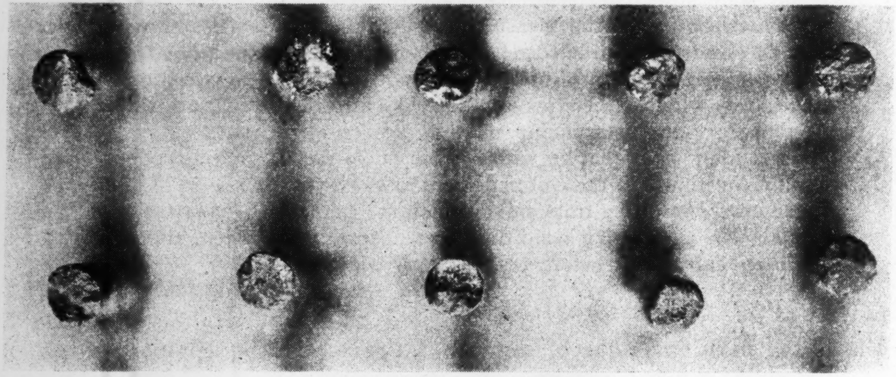

FIG. 1.—Fatigue Fractures in Bead Wire from a Passenger Car Tire that Failed in Service.

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[3] C. L. Beal, "Anode Process for Rubber Articles and Coatings," *Industrial and Engineering Chemistry*, Vol. 25, p. 609 (1933).

[4] Leonard Church, "Rubber Coatings for Abrasion and Corrosion Resistance," *Chemical and Metallurgical Engineering*, Vol. 40, p. 467 (1933).

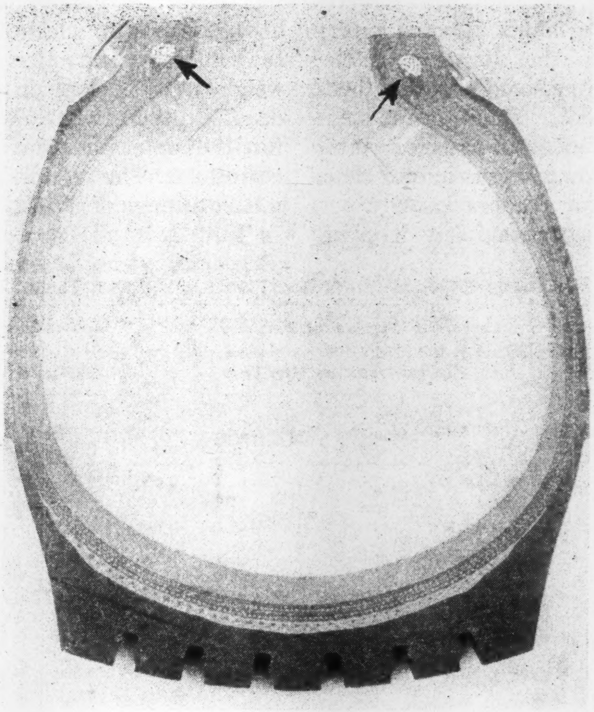

FIG. 2.—Bead Wire in the Tire.

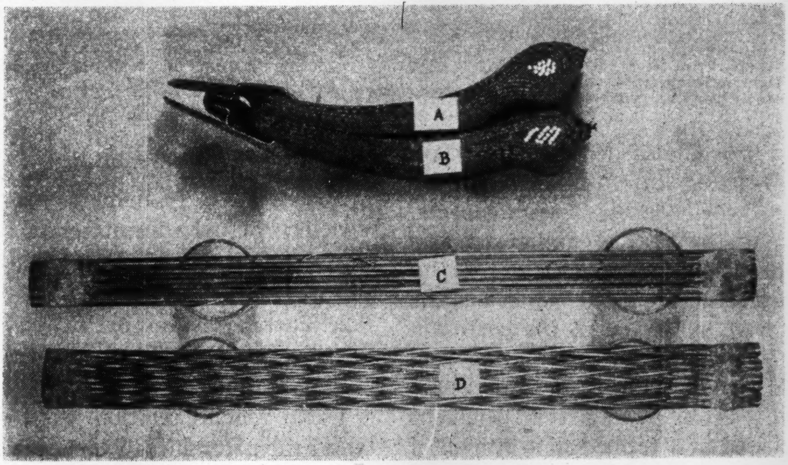

FIG. 3.—Commercial Types of Bead Wire Construction.

A. Abbott. B. Alderfer (Westless).
C. Tape. D. Braid.

DeCroly.[5] The test samples are attached to a carrier which rises and lowers every 15 min., thus immersing the materials in solutions and exposing them to the atmosphere. The aqueous solutions are made up with distilled water which is replenished, from time to time, to compensate for evaporation.

The test specimens were copper-zinc and bronze-plated bead wire, 0.037 in. in diameter, carefully prepared by cleaning with carbon tetrachloride, weighed to the nearest milligram, and suspended from the movable carrier by paraffined strings. About 7.5-in. lengths of wire were subjected to the corrosion tests. Two samples of each plating were tested in a given solution and the results averaged. After testing, the specimens were washed and rubbed in a stream of tap water to remove all possible adhering deposits, cleaned with carbon tetrachloride and alcohol, and again weighed. The loss in weight in ten days' exposure was taken as the basis for the determination of the rate of corrosion. The results of the intermittent immersion tests are summarized in Table I.

TABLE I.—RESULTS OF INTERMITTENT IMMERSION TESTS.

		Copper-Zinc Plated Bead Wir[d]				Bronze Plated Bead Wire[e]			
Solution	pH Value	Loss, g. per sq.cm. per yr.[a]	Physical Properties After Tests			Loss, g. per sq.cm. per yr.[a]	Physical Properties After Tests		
			Tensile Strength, psi.	Cold Bends[b]	Surface Appearance		Tensile Strength, psi.	Cold Bends[b]	Surface Appearance
NH_4OH, 3%....	9.1	0.084	276 000	4.0	Dark blue, glossy, and smooth	0.006	275 000	4.0	Bright iron surface
NaCl, 2%.......	7.1	0.064	273 000	3.0	Rough and somewhat pitted	0.062	239 000	3.0	Very rough and pitted
Uric acid, 5%...	6.7	0.006	277 000	3.5	Copper plating dulled and attacked in spots	0.100	265 000	3.0	Rough, indicating heavy attack on steel wire
Hippuric acid, 5%...........	5.0	0.160	273 000	3.5	Bright iron surface	0.059	275 000	4.0	Smooth iron surface
Na_2S, 7%.......	10.6	0.009	277 000	4.0	Smooth, brassy, green tinge	0.006	275 000	4.5	Steel blue
H_2SO_3 (SO_2, 3%)		The wires were completely dissolved within 48 hr.							
Combination of all............	7.1	0.048	273 000	2.5	Rough and tending to pit	0.009	275 000	2.5	Bright in spots but deeply attacked elsewhere
Distilled water..	6	0.009	276 000	3.5	Copper plating bright but dulled in spots	0.028	269 000	2.5	Slightly roughened
Average[c].............		0.054	275 000	3.4		0.039	269 000	3.4	
Original........................			280 500	4.0			275 000	4.5	

[a] The average of two determinations.
[b] Cold, right angle, reverse bends.
[c] Does not include results of the H_2SO_3 tests.
[d] Zinc by "hot dip" plus copper by electrodeposition.
[e] Bronze by "displacement."

General Conclusions of the Corrosion Tests:

1. The plated wire materials retained to a large extent their original tensile and ductile properties.
2. There was no indication of embrittling effects which might indicate intercrystalline corrosion.
3. No general relation was found between the pH values of the solutions and the loss in metal.
4. The loss in metal appeared to be

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[5] Colin G. Fink and C. M. DeCroly, "The Corrosion Rate of Ferro-Nickel Alloys," *Transactions*, Am. Electrochemical Soc., Vol. 56, p. 239 (1929).

more a function of the solubilities of the plating materials than of their relative positions in the electromotive series.

5. Low losses in metal may be attributed, to some extent, to the formation of protective films and to the fact that these films remained intact under the unstressed condition of the wire.

The tensile and ductile properties of the wire were not seriously impaired; with the exception of one solution, which dissolved the wire, only relative differences were exhibited by the solutions in their chemical attacks. There is no way of predicting how, and to what extent, aqueous solutions may come in contact with bead wire under service conditions. It certainly would not be under the unstressed condition of the intermittent immersion test. The question now arises as to what extent protective films may develop under the "no load" conditions of this test. Would such protective films be prevented, or else destroyed, under the variable loading conditions of service? It was noted that some of the specimens tended to corrode and pit, while others exhibited smooth surfaces even though they had undergone equal losses in metal. It remained to be determined whether such conditions would obtain, had these plated materials been subjected simultaneously to chemical attack and variable stress.

Corrosion-Fatigue Testing of Wire:

Fatigue tests were decided upon as the best method for determining the relative behaviors of plated steel wire materials in corrosive environments. The rotating-wire arc fatigue machine, described before the Society[6] in 1935 was used for these tests. Essentially this method consists in bending a wire test specimen to a curve by means of two inclined babbitt metal guides and rotating this wire from one end by a motor. The other end of the wire is left free to slide longi-

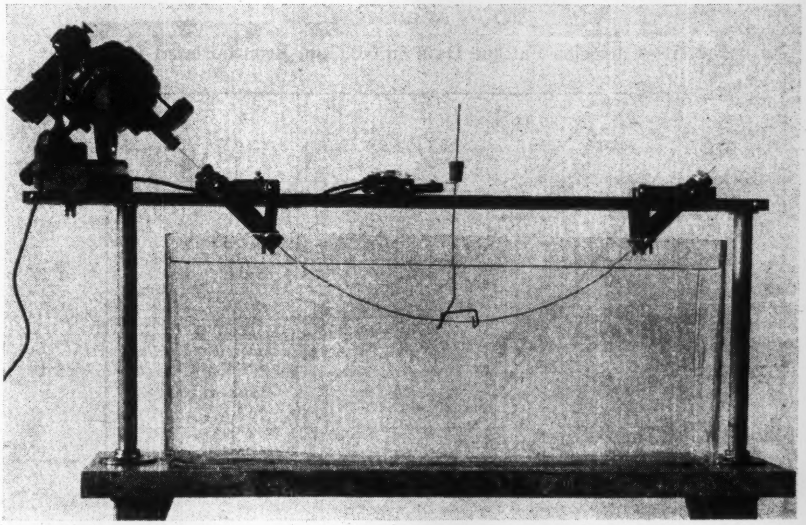

FIG. 4.—Rotating-Wire Arc Fatigue Machine (Author's Design).
NOTE.—Exposure of 15 sec. with machine operating at 6000 r.p.m.

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[6] J. N. Kenyon, "The Rotating-Wire Arc Fatigue Machine for Testing Small Diameter Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 156 (1935).

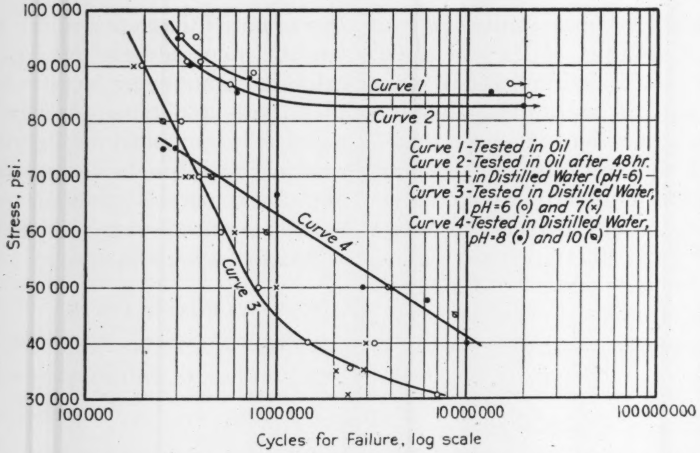

FIG. 5.—Corrosion-Fatigue Data on 0.037-in. Bronze-Plated Wire.

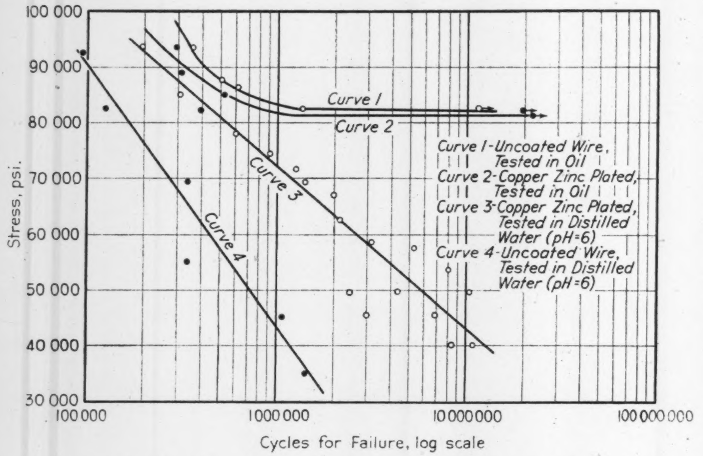

FIG. 6.—Corrosion-Fatigue Data on 0.037-in. Wire.

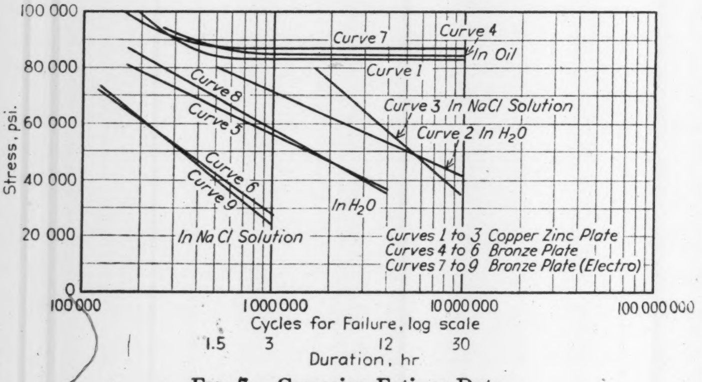

FIG. 7.—Corrosion-Fatigue Data.

tudinally in its guide and, in so doing, the test specimen *automatically* assumes the curvature of the circular arc. Some 15 in. of wire are thus subjected to equal stress reversals with fatigue failure occuring at the weakest point within this length. The rotating arc dips into an oil bath which dampens out vibrational effects. This dampening method offers a further advantage for making corrosion-fatigue tests wherein the oil bath is replaced by aqueous solutions, see Fig. 4.

TABLE II.—ENDURANCE DATA OBTAINED ON BEAD WIRE WITH THE ROTATING-WIRE ARC FATIGUE MACHINE.

	Coating	Tested in	pH Value	Endurance Limit, psi.	Original Tensile Strength, psi.	Ratio, Endurance Limit to Tensile Strength
Fig. 5						
Curve 1	Bronze[a]	Oil		85 000	275 000	30.9
Curve 2		(Oil)[c]		82 500		30.0
Curve 3		Distilled water	6 and 7	30 000		11.0
Curve 4		Distilled water	8 and 10	40 000		14.5
Fig. 6						
Curve 1	Plain	Oil		82 500	326 000	25.3
Curve 2	Copper-zinc[e]	Oil		81 500	280 500	29.1
Curve 3	Copper-zinc[e]	Distilled water	6	43 000	280 500	15.4
Curve 4	Plain	Distilled water	6	(20 000)	326 000	[d]
Fig. 7						
Curve 1	Copper-zinc[e]	Oil		83 000	294 500	28.2
Curve 2		Distilled water	6	42 000		14.3
Curve 3		NaCl solution		36 000		12.3
Curve 4	Bronze[a]	Oil		85 000	297 500	28.6
Curve 5		Distilled water	6	29 000		9.8
Curve 6		NaCl solution				[d]
Curve 7	Bronze[b]	Oil		87 000	292 500	32.7
Curve 8		Distilled water	6	24 000		8.2
Curve 9		NaCl solution				[d]

[a] Plated by displacement.
[b] By electro-deposition.
[c] After storage in distilled water for 48 hr.
[d] Not determined.
[e] Zinc by "hot dip" plus copper by electrodeposition.

Discussion of the Corrosion-Fatigue Data:

Corrosion-fatigue data obtained by the arc-method are summarized in Figs. 5, 6, 7 and in Table II.

Figure 5 gives comparative fatigue values on bronze plated wire resulting from tests made under the following conditions:

1. Tested in oil (non-corrosive).
2. Tested in oil after storing for 48 hr. in distilled water, (pH = 6).
3. Tested in distilled water, (pH = 6, 7, 8 and 10), where the exposure to water did not exceed 30 hr.

Distilled water was selected for the corroding medium since it was believed that the corrosive effect could be somewhat controlled by the pH value. The wire specimens, while stored in water, developed a feathery brown precipitate which indicated that the steel had undergone chemical attack through pinhole openings in the plating. However, these stored specimens did not show much loss in the endurance properties. It may be that protective films developed at the pinhole openings in the plating and so arrested any serious pitting effect. The specimens tested directly in distilled water exhibited a most pronounced lowering in the endurance properties—a loss in the order of 65 per cent, after 10,000,000 cycles of stress. It is likely that protective films, which tend to form at the pinhole openings in the bronze plating, were prevented, or else destroyed, under variable stress conditions of this corrosion-fatigue test. As a result the steel wire underwent an excessive pitting effect at every pinhole opening in the plating, to the serious detriment of the endurance properties. Such corrosion-fatigue data bring out forcibly the unreliability of corrosion or fatigue tests made separately for indicating service qualities under even mildly corrosive conditions. (Compare curves 3 and 4 with 1 and 2.)

Curves 3 and 4 of Fig. 5 represent the data obtained when the specimens were tested in a water bath with pH values of 6, 7, 8, and 10. The specimens tested in the medium with a pH value of 6 and 7 show a more abrupt drop in endurance qualities than does the fatigue data obtained in the medium with a pH value of 8 and 10. It is possible that with higher oxygen concentration the oxidizing effect on the film tended to repair breaks in it.[7]

Figure 6 shows fatigue values obtained for uncoated and copper-zinc coated wire tested in oil and in distilled water (pH = 6).

The fatigue limits for 10,000,000 cycles of stress of the two materials were practically identical under the non-corrosive test conditions of the oil bath. In the distilled water bath, copper-zinc plated wire was vastly superior to unplated wire; it was also much superior to the bronze plated wire (compare Fig. 5).

Due to the importance of the corrosion-fatigue data revealed in Figs. 5 and 6, another series of tests was conducted in order to bring out further information on plating technique and materials, Fig. 7. The tests, in oil, distilled water (pH = 6) and 3 per cent salt solutions, were made on the following materials: (1) copper-zinc plated, (2) bronze plated by displacement, and (3) bronze plated by electro-deposition.

When subjected to stress-reversal at 40,000 psi., the copper-zinc plated wire withstood nearly 10,000,000 cycles of stress both in distilled water and in salt solution, whereas the bronze plated wire materials withstood only 3,000,000 cycles of stress in distilled water and 500,000 cycles of stress in salt solution, Fig. 7. Even more significant was the ductile properties of the three plated wire materials after the corrosion-fatigue tests. The copper-zinc plated wire could be bent into a helix around a $\frac{1}{8}$-in. diameter rod while the two bronze-plated wires were easily broken up with the fingers. A microscopic examination revealed numerous fatigue cracks in the bronze-plated materials which were often only 0.02 in. apart. The multiplicity of these cracks may well account for the embrittled condition of these bronze-plated wire materials, Figs. 8 and 9.

The embrittling effect produced by aqueous solutions on bronze-plated wire subjected to a corrosion-fatigue test may be explained by the relative positions of metals in the electromotive series. Bronze plating (Cu + 0.1 Sn), is more noble than steel. In aqueous solution, steel must be anodic to bronze because of minute imperfections in platings. These pinhole imperfections dilate under the action of variable stress, and, as a result, the steel undergoes an excessive pitting attack. Actually the steel, being anodic, is more rapidly attacked in spots than if the bronze plating were not present. This pitting effect, if the assumptions are correct, must proceed at a very rapid rate since the more highly stressed bronze plated wire specimens actually became embrittled within 30 min. Similar corrosion-fatigue tests on unplated wire did not reveal pronounced embrittlement. The tested wire could not be broken up with the fingers and, except for an occasional break, could be bent into a helix around a $\frac{1}{8}$-in. diameter rod.

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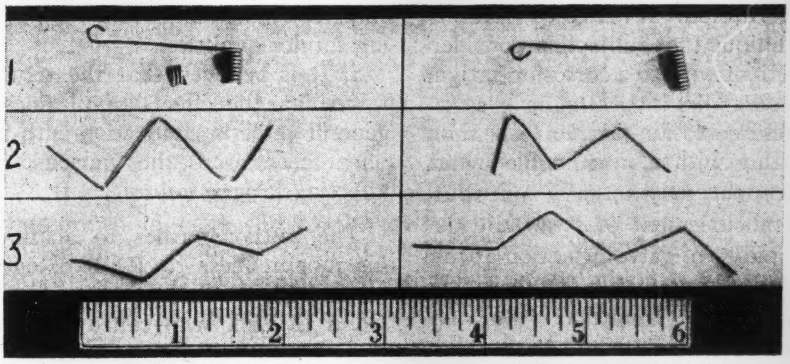
[7] E. C. Groesbeck and L. J. Waldron, "Oxygen as a Factor in Submerged Corrosion," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 279 (1931).

The fact that copper-zinc plated wire withstood these corrosion-fatigue tests so well is likewise explainable by reference to the electromotive series. Zinc is less noble than steel; in an aqueous solution the zinc is anodic and tends to go into solution and so protects the steel. As a result the steel was subjected to less pitting attack at pinhole openings in the platings since it was cathodic to the zinc at these points.



In distilled water. In salt solution.

FIG. 8.—Showing Bronze-Plated Bead Wire Embrittled by Corrosion-Fatigue.

1. Copper-zinc plated wire.
2. Bronze plated by displacement.
3. Bronze plated by electro-deposition.

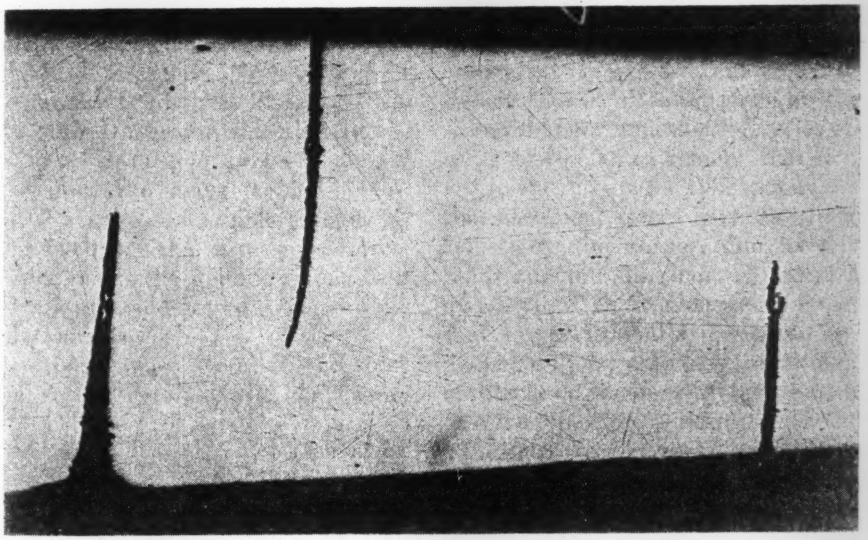


FIG. 9.—Microscopic Appearance of Embrittled Bronze-Plated Wire (× 60).

Conclusions:

1. A small wire fatigue machine, developed for testing wire in lengths sufficient for integrating the effects of various surface irregularities, has proved to be

most effective for making corrosion-fatigue tests using aqueous solutions.

2. Embrittled steel wire, often encountered in service, is probably due to the formation of fatigue cracks. Such conditions are only revealed by a laboratory technique that subjects a considerable length of wire to a corrosion-fatigue test.

3. These corrosion-fatigue tests show that plating with a more noble metal, such as copper, may actually contribute to the embrittlement of steel wire due to the rapidity of galvanic action at pinhole points of imperfection in the plating. No embrittled condition was encountered in wire that had been galvanized before plating with copper.

4. It is shown that static corrosion and static physical tests, often relied upon to determine the probable service qualities of plated steel wire, are inadequate. Only fatigue tests, wherein there is simultaneous corrosive attack, are sufficiently discriminatory for determining service qualities.

5. It is believed that the results obtained on the effects of platings have general value in connection with fatigue phenomena and the various factors affecting fatigue failure.

The author wishes to acknowledge his indebtedness to R. R. Rogers for advice on the corrosion tests; and to Colin G. Fink who called attention to the research possibilities offered by bead wire and contributed most generously with constant advice to the study.

DISCUSSION

Mr. Peter R. Kosting[1] (*in letter to author*).—Before accepting Mr. Kenyon's explanation of the reason for the benefits of the copper-zinc coating on wire, as described in page 713 of the paper, I would appreciate being informed further regarding the following points:

1. Were the copper and zinc co-deposited to form a brass or were they deposited separately?
2. What is the approximate composition of the copper-zinc plate?
3. Were the bronze and the copper-zinc plates of equal thickness and of equal freedom from porosity prior to test?

Mr. R. E. Peterson[2] (*by letter*).—If one considers bead wires located at the rim of a tire, it is not immediately obvious why a fatigue problem should exist. Since the main possibility appears to be due to variable tension resulting from variable centrifugal force, and since it was the writer's feeling that such stresses would not be large, a rough calculation was made.

The approximate value of the force tending to rupture the tire can be expressed as $T = \frac{WV^2}{2g\pi r}$. Estimating weights and radii, a value of approximately 500 lb. is obtained for 50-mph. car speed. Assuming that because of large difference of moduli the wires carry most of the load, and that there are 18 wires of 0.037-in. diameter in each bead, a stress of about 13,000 psi. is obtained. Comparing this with the fatigue data obtained by the author, a pulsating stress of 0 to 13,000 psi. would correspond in terms of fatigue failure to an alternating stress of about ±8000 psi.[3] It is obvious that, since the wire has an endurance limit of better than ±80,000 psi., failure will not take place unless a drastic effect, such as corrosion fatigue, occurs. Even so, if we consider such a drastic effect as given by curve 4 of Fig. 6, we see that at 8000 psi. the number of cycles required would be of the order of several million.[4] It is difficult to see how one could obtain more than 10,000 cycles of equivalent speed variation in the usual life of a tire. Examination of Fig. 6 shows that for a number of cycles of the order of 10,000, extremely high stresses would be required. It is possible that the additional forces such as are mentioned by the author, due to uneven roads and rounding curves, are of high magnitude. It would seem, however, that the occasional failures can be explained only by extremely abnormal operating conditions, or by defective wire, or a combination of both.

Although the paper is valuable quite apart from the bead wire application, the author has evidently given this some thought, and some further remarks along this line would be of interest.

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[1] Chemical Engineer, Watertown Arsenal, Watertown, Mass.

[2] Manager, Mechanics Division, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[3] The effect of steady stress due to inflation pressure would not alter this statement materially.

[4] One might expect a lower curve at the slow frequency of service cycles, but an examination of McAdam's data leads one to conclude that a large difference would not be obtained. D. J. McAdam, Jr., "Stress-Strain-Cycle Relationship and Corrosion-Fatigue of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 248, Fig. 1 (1926).

MR. JOHN N. KENYON[5] (*Author's closure*).—In reply to Mr. Kosting's questions, the following may be stated:

1. The copper and zinc were applied separately: the zinc by the "hot dip" process, the copper by electrodeposition.

2. The copper-zinc plating is from commercially pure materials. The bronze plate is commercially pure copper containing a trace of tin (approximately 0.1 per cent).

3. The bronze plate is put on by the displacement process and is relatively thin; the copper-zinc plate is thicker.

The main purpose of the study was to compare the *service* qualities of two bead wire materials as commercially produced; both materials are being extensively used today for automobile tire reinforcement. The corrosion-fatigue technique, developed in this study, can be equally well used to compare platings of equal thickness and freedom from porosity.

Mr. Peterson is correct in the statement that bead wire failures appear to be unlikely when considered from an analysis of the variable forces acting on the tire. However, it would seem that the following are the controlling considerations:

1. Due to mechanical injury or the slow oxidation of rubber with prolonged service, water often finds its way into the beads of a tire and corrodes the wire.

2. The so-called static tests (tension, torsion, cold bends and corrosion) have heretofore been relied upon to gage the physical properties of bead wire, but they do not differentiate the relative endurance qualities of these materials in any significant manner.

3. The corrosion-fatigue test, developed in this study for wire, reveals marked differences in the relative endurance qualities of bead wire. It was shown that one plated wire material retained a high degree of ductility, whereas another rapidly became embrittled and could be broken up with the fingers.

4. Bead wire which may become embrittled in service—a condition largely due to corrosion-fatigue—has lost its shock resisting properties and the ability to withstand a sudden overload application.

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[5] Instructor in Civil Engineering, Columbia University, New York, N. Y.

FATIGUE TESTS ON ZINC-COATED STEEL WIRE

By D. G. Watt[1]

Synopsis

Reversed bending fatigue tests were made on zinc-coated steel wires of three strengths. In general, the endurance limit increased with the tensile strength while the endurance ratio diminished. Wire from the same reels tested in the lead-annealed and hot-dip galvanized conditions showed the deleterious effect of the coating process on the fatigue properties. No perceptible difference was observed between the endurance limits of wire from corresponding reels hot-galvanized with four and three-and-one-half dip coatings. Reduction by drawing, within the range investigated, increased the fatigue resistance of the wires both in the lead-annealed and zinc-coated conditions.

Hot-dip zinc coatings developed, under reversed stressing, closely spaced hair cracks perpendicular to and terminating at the surface of the steel. In electrodeposited zinc coatings no evidence of such cracks could be found after similar treatment. The results obtained on defective wires, containing surface seams or nonmetallic inclusions, demonstrated the utility of the fatigue test for detecting faulty material. No definite correlation could be observed between the data furnished by the ordinary bend and twist tests, as carried out at the mill, and the endurance limits of the wires.

The progressive failure of overhead conductor cables due to aeolian vibrations presents a serious problem to engineers responsible for the design and maintenance of power-transmission lines. A great amount of study has been devoted to the cause and nature of conductor vibration and to the devising of mechanical dampers to suppress it below a harmless limit.

Coincident with the advances which have been made in restricting the intensity of cable vibration to the point where indefinitely long or at least extended conductor life will result, efforts have been made to obtain conductor materials with the best fatigue properties and to ensure that these properties are maintained during manufacture by checking from time to time the endurance limit of the wire from which the conductor is being fabricated.

The simple reversed bending fatigue test, while it does not duplicate the complex stress conditions to which the wires in a vibrating cable are subjected in the field, furnishes the most practical method of measuring the relative ability of materials to withstand vibration failure. For application of wire in overhead conductors, where aeolian vibrations may occur, the results of fatigue tests on the material have more significance than much of the data obtained from the standard or commonly prescribed wire-quality tests. Measurements of tensile strength, elastic limit, and ductility still constitute the basic

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[1] Assistant Testing Engineer, The Hydro-Electric Power Commission of Ontario, Toronto, Ont., Canada.

TABLE I.—SUMMARY OF TEST DATA.

Grade	Coil Number	Wire Diameter, in. (Steel)	Composition, per cent				Condition	Weight of Zinc Coating, oz. per sq. ft.	Tensile Strength, psi.	Elongation in 10 in., per cent	Twists to Failure, Length = 100 D		Bends to Failure, 180 deg., r = 0.187 in.		Modulus of Elasticity, psi.	Endurance Limit, psi.	Endurance Ratio, per cent
			Carbon	Manganese	Phosphorus	Sulfur					Number	Average Variation, per cent	Number	Average Variation, per cent			
220,000 to 250,000 psi...	A 6A1-083-286	0.082	0.60	0.98	0.028	0.028	Lead-annealed	....	232 500	5.1	26.4	3.0	17.0	0.0	28 800 000	73 200	31.4
	A 6A2 083-272	0.082							229 000	5.0	30.5	1.5	17.0	0.0	29 200 000	73 000	31.8
	A-7A1-104-276	0.103	0.75	1.10	0.029	0.030	Lead-annealed	....	258 200	5.1	24.1	1.0	10.8	2.8	29 000 000	71 500	27.6
	A 7A2-104-280	0.103							255 800	5.0	27.4	1.0	8.8	3.4	28 700 000	73 800	28.9
	A-7A1-125-282	0.123							227 000	5.4	27.4	1.0	11.6	3.4	27 800 000	65 000	28.6
	A-7A2-125-284	0.123							225 800	5.3	27.2	1.0	8.8	4.2	28 000 000	67 200	29.8
	A-6G1-083-287	0.082	0.60	0.98	0.028	0.028	Hot-dip galvanized	0.087	227 000	5.3	26.5	2.0	13.4	3.7	28 700 000	58 200	25.6
	A-6G2-083-273	0.082						0.088	226 500	5.2	23.8	3.0	11.8	2.5	28 700 000	58 500	25.8
	A-7G1-104-277	0.103	0.75	1.10	0.029	0.030	Hot-dip galvanized	0.95	248 000	5.6	23.1	3.0	11.0	0.0	28 900 000	60 500	24.4
	A-7G2-104-281	0.103						0.92	248 000	5.7	23.1	1.0	9.0	0.0	28 800 000	60 500	24.4
	A-7G1-125-283	0.123						1.05	223 000	5.5	27.6	3.0	7.0	0.0	29 200 000	54 200	24.3
	A-7G2-125-285	0.123						1.01	224 000	5.5	28.2	1.0	9.0	0.0	28 400 000	54 100	24.2
	C-7E -083-200	0.080	0.73	0.67	....	....	Electrogalvanized	0.92	255 800	5.2	26.2	15.0	16.0	0.0	28 900 000	72 700	28.4
	C-7E -104-201	0.101	0.74	0.63	....	....	Electrogalvanized	0.94	240 100	5.4	26.2	13.0	11.0	0.0	28 500 000	66 400	27.6
	C-7G -104-202	0.101	0.74	0.71	....	....	Hot-dip galvanized	1.08	230 100	5.2	12.8	43.0	9.3	4.3	29 100 000	57 100	24.7
	C-7E -125-203	0.122	0.67	0.72	....	....	Electrogalvanized	0.90	234 900	5.3	17.0	27.0	7.0	0.0	28 500 000	58 600	24.9
170,000 to 190,000 psi...	B-5A1-083-236	0.080	0.50	1.06	0.025	0.023	Lead-annealed	....	181 000	6.4	37.9	1.7	17.0	0.0	29 500 000	69 500	38.4
	B-5A2 083-241	0.080							183 000	6.9	35.3	3.4	17.4	2.9	29 400 000	73 000	39.9
	B-5A3 083-246	0.080							187 700	6.0	36.0	0.0	19.0	0.0	29 600 000	69 000	36.8
	B 5A4-083-251	0.080							186 200	6.0	34.9	2.3	19.2	1.6	29 500 000	74 100	39.8
	B-5A1-104-200	0.101							170 200	6.9	33.4	4.1	13.1	0.0	29 600 000	67 200	39.4
	B-5A2-104-224	0.101							175 800	6.9	31.5	3.5	13.4	3.7	29 200 000	65 700	37.4
	B-5A3-104-228	0.101							178 800	7.0	35.0	6.3	14.0	0.0	29 000 000	64 500	36.1
	B-5A4-104-232	0.101							172 800	7.3	33.4	6.9	11.8	2.5	29 000 000	66 000	38.2
	B-5A1-125-204	0.117							171 200	6.6	30.0	1.3	11.0	0.0	28 500 000	61 400	35.8
	B-5A2-125-208	0.117							176 100	6.4	30.4	1.6	11.8	2.5	28 700 000	61 100	34.6
	B-5A3-125-212	0.117							177 100	6.4	31.0	0.0	9.0	0.0	28 200 000	62 600	35.3
	B-5A4-125-216	0.117							178 000	6.6	33.0	0.0	11.0	0.0	28 700 000	65 100	36.6

	B-5G1-083-238	0.080	0.50	1.06	0.025	0.023	Hot-dip galvanized	0.88	179 200	6.1	23.8	3.2	11.2	2.7	29 800 000	50 800	28.4
	B-5G2 083-243	0.080						0.89	181 200	6.0	23.5	2.2	15.0	0.0	29 500 000	52 500	29.0
	B-5G3 083-248	0.080						0.85	182 000	6.0	23.2	1.4	12.2	2.4	29 600 000	52 600	28.9
	B-5G4-083-253	0.080						0.90	182 800	6.1	22.9	2.8	15.0	0.0	29 200 000	53 000	29.0
	B-5G1-104-222	0.101						0.93	170 400	6.5	22.0	0.0	11.0	0.0	29 300 000	50 900	29.8
	B-5G2-104-226	0.101						1.15	168 000	6.6	21.6	2.2	11.2	2.7	29 200 000	47 500	28.3
	B-5G3-104-230	0.101						0.91	175 400	7.3	22.4	2.1	13.0	0.0	29 200 000	51 000	29.0
	B-5G4-104-234	0.101						0.94	170 000	7.1	22.4	2.2	11.0	0.0	29 000 000	48 500	28.6
	B-5G1-125-206	0.117						0.96	176 100	6.3	29.2	1.1	11.0	0.0	28 300 000	48 500	27.5
	B-5G2-125-210	0.117						0.81	178 000	6.5	29.2	1.1	11.0	0.0	28 600 000	48 300	27.1
	B-5G3-125-214	0.117						0.98	176 000	6.2	30.2	3.2	9.0	0.0	28 200 000	46 000	26.1
	B-5G4-125-218	0.117						0.90	178 100	6.0	27.2	1.2	9.0	0.0	28 300 000	48 00	26.9
	D-5E -104-259	0.100	0.51	1.07	0.014	0.024	Electrogalvanized	0.90	189 800	7.2	29.5	1.7	11.0	0.0	28 700 000	56 200	29.6
	D-5E -104-260	0.098						2.03	191 400	7.3	29.6	1.6	10.8	2.7	29 600 000	60 500	31.6
	D-5E -104-261	0.095						2.30	189 700	7.4	27.6	2.4	9.0	0.0	29 000 000	58 500	30.8
	E-6E -083-221	0.080	0.61	0.78	....	....	Electrogalvanized	1.05	189 500	6.3	30.6	9.0	8.3	7.2	28 500 000	65 000	34.3
	E-6E -104-223	0.101	0.62	0.87	....	....	Electrogalvanized	0.87	171 700	7.8	31.0	5.5	8.0	0.0	28 400 000	62 000	36.1
	E 6G -104-225	0.101	0.60	0.81	....	....	Hot-dip galvanized	1.10	175 000	7.2	17.0	7.5	7.8	3.9	28 500 000	54 800	31.3
	E-6E -125-227	0.122	0.61	0.92	....	....	Electrogalvanized	1.03	171 300	7.5	28.0	5.5	6.0	0.0	29 000 000	58 300	34.0
110,000 to 120,000 psi...	F-2E -083-263	0.080	0.21	0.46	....	....	Electrogalvanized	0.95	118 000	6.4	34.7	9.8	18.0	6.6	28 400 000	50 600	42.9
	F-2E -104-264	0.101	0.19	0.42	....	....	Electrogalvanized	1.05	116 500	8.5	35.5	1.5	10.0	0.0	29 100 000	47 900	41.1
	F-2G -104-265	0.101	0.17	0.50	....	....	Hot-dip galvanized	1.01	112 800	8.2	19.8	3.0	8.9	2.2	28 600 000	43 100	38.2
	F-2E -125-266	0.122	0.20	0.49	....	....	Electrogalvanized	0.94	110 100	7.8	17.4	3.0	8.5	7.0	29 200 000	45 500	41.3

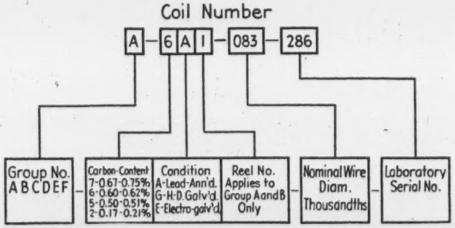
Coil Number
A
6
A
1
083
286
Group No. ABCDEF
Carbon-Content 7-0.67-0.75% 6-0.60-0.62% 5-0.50-0.51% 2-0.17-0.21%
Condition A-Lead-Ann'd. G-H.D.Galv'd. E-Electro-galv'd.
Reel No. Applies to Group A and B Only
Nominal Wire Diam. Thousandths
Laboratory Serial No.

FIG. 1.—Key to Coil Numbering System.

requirements of the majority of wire specifications. In spite of the fact that such data are essential to the designer and in many instances are related to service life, it is common knowledge that the results fail to reveal whether the material has the required fatigue strength for applications in which vibratory stressing may occur.

The work recorded in this paper represents a part of the Hydro-Electric Power Commission's researches on the fatigue properties of conductor materials, namely, that dealing with zinc-coated steel wire of the type from which transmission line ground wire cables are stranded.[2]

Briefly, the objects of the investigation were as follows:

1. To compare the endurance limits of zinc-coated steel wires of the following grades:

220,000 to 250,000 psi.
170,000 to 190,000 psi.
110,000 to 120,000 psi.

2. To determine the extent to which the hot-dip galvanizing process affects the endurance limit of steel wire in the lead-annealed condition.

3. To determine, within a specified range, the influence of the thickness of zinc coating on the endurance limit.

4. To determine whether the results of the ordinary quality tests, as carried out at the mill, namely, tensile strength, percentage elongation, bends and twists to failure, give any indication of the fatigue properties of the wire.

5. To supplement physical tests by microscopic examinations of the wires and their coatings to determine, where possible, the factors which are deleterious to the fatigue properties of the material.

Materials:

The investigation was carried out on wires classified on the basis of tensile strength in Table I. The tabulated data include the condition in which the wire was tested, the chemical composition, diameter, and weight of zinc coating. The key to the sample numbering system is explained in Fig. 1.

All coils of 170,000 to 190,000 psi. grade wire in group B were drawn from the same melt of steel. Samples were obtained from four reels of wire diameters 0.080, 0.101, and 0.117 in. Part of each reel was withdrawn from the continuous process after lead-bath annealing. The remainder was subjected to the complete coating process, including lead-bath annealing, pickling, and hot-dip galvanizing.

The 220,000 to 250,000 psi. grade wires in group A were drawn from two melts of steel, the one of lower carbon content being used for the 0.082-in. diameter wire. The sample coils were obtained from two reels of each diameter. As in the case of the wires in group B, a portion of each reel was lead-annealed and the remainder hot-dip galvanized.

All of the wires in group D and some of the wires in groups E, F, and G were zinc coated by electrolytic processes.

Test Methods:

The fatigue data presented in this paper were obtained from tests carried out on two Haigh-Robertson wire fatigue testing machines, one of which is shown in Fig. 2. Since these testing machines, or slight modifications, are in general use and have been described in detail elsewhere, it is only necessary to outline briefly the principle of operation here.[3]

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[2] Ground wire cables are seven-strand galvanized steel cables strung between transmission line towers for protection against lightning.

[3] "Fatigue Testing Machine for Wire," *Engineering*, Vol. 138, August 10, 1934, p. 139.

In Fig. 2 it will be observed that one end of the wire specimen under test is gripped in the chuck on the motor shaft. The other, fitted with a small cup-faced sleeve, runs on a ball thrust bearing mounted in a movable tailstock on the bed of the machine. The headstock, including the driving motor and chuck, is free to turn on a vertical axis coincident with the chuck jaws. By means of a fine screw adjustment, the thrust bearing on the tailstock may be moved towards the headstock and the wire specimen thus given any desired degree of deflection as a strut and simultaneously rotated by the motor about the neutral axis.

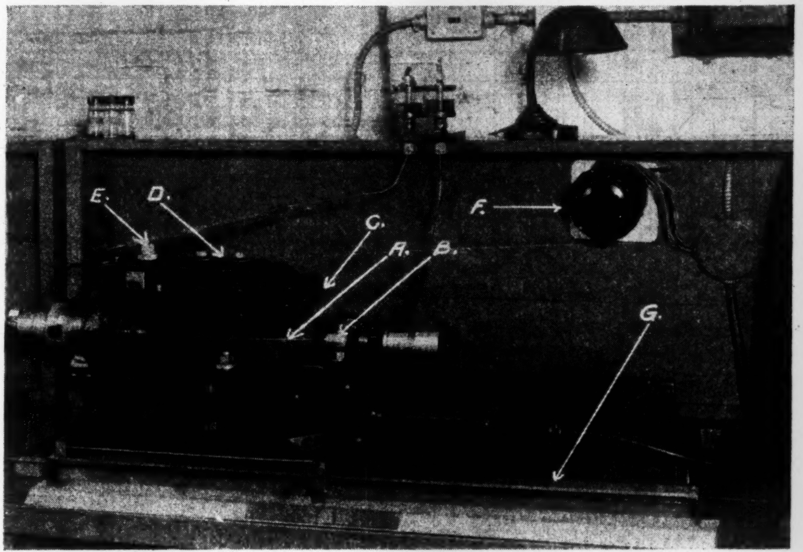

FIG. 2.—Haigh-Robertson Fatigue Testing Machine.

A - Wire specimen.
B –Tailstock.
C – Angular scale.
D—Headstock.
E—Vertical headstock bearing.
F –Speed control.
G—Specimen length gage.

If the modulus of elasticity of the wire is known, the maximum stress in the specimen, which occurs at mid-length, may be calculated from its length-diameter ratio and the angle of inclination at the free ends. This angle is read directly from the vernier angular scale attached to the swinging headstock. In the case of the zinc-coated wires the length-diameter ratio was based on the diameter of the steel. The principle of load measurement employed in the Haigh-Robertson machine assumes that the wire specimen was originally straight. If this requirement is not fulfilled and the wire contains any small kinks, twists of long radius bends, secondary vibrations will be set up in the rotating specimen and it will not conform to the smooth curve on which the stress calculations are based.

The wire samples, as received, contained the natural curvature of the coil produced by winding on the take-ups in the galvanizing plant. Straightening of the specimens was first carried out by pulling each in a testing machine to the so-called yield point determined by the drop of the beam. This method, which was originally suggested by the makers of the Haigh-Robertson machine, proved fairly satisfactory for 170,000 to 190,000 psi. grade wire, although at times supplementary hand straightening was required.[4] In the case of the harder wires it was found that straight specimens could be obtained more conveniently by passing the wire through a small hand-operated straightening machine. Accordingly, after all of the tests had been completed on groups A, B, and D, the remaining tests were carried out on machine-straightened specimens.

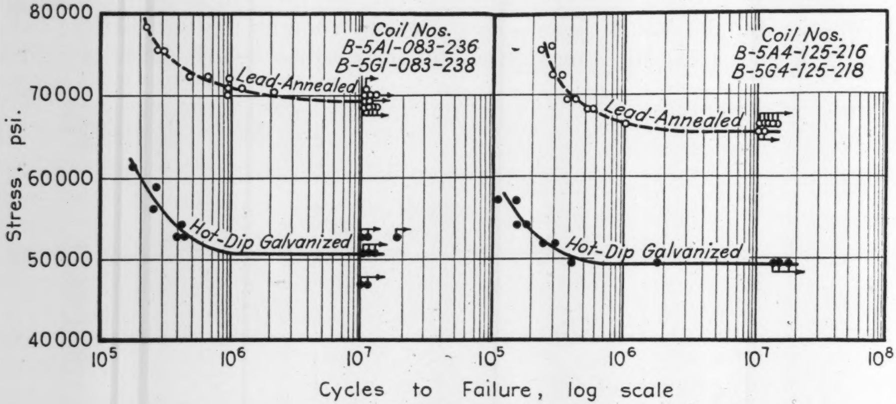

FIG. 3.—*S-N* Curves on 170,000 to 190,000 psi. Grade Lead-Annealed and Hot-Dip Galvanized Wire.

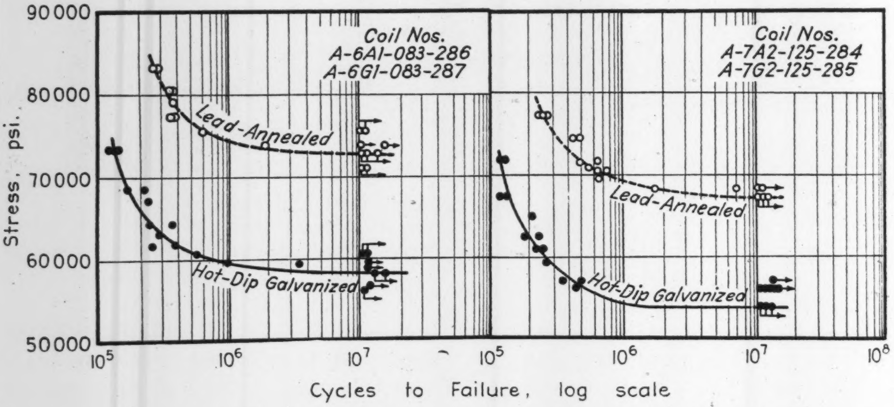

FIG. 4.—*S-N* Curves on 220,000 to 250,000 psi. Grade Lead-Annealed and Hot-Dip Galvanized Wire.

The testing speed used was of the order of 6000 to 7000 rpm. In some of the tests higher and lower speeds were employed to eliminate specimen vibration.

Modulus of elasticity determinations were carried out on at least two wire specimens from each coil, strain measurements being recorded over a 10-in. gage length with an Anderson extensometer.[5] The samples for modulus of elasticity tests were straightened by the same methods as those employed for straightening the fatigue specimens.

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[4] C. P. Wampler and N. J. Alleman, "Fatigue Tests of Wire," ASTM BULLETIN, No. 101, December, 1939, p. 13.

[5] H. A. Anderson, "Tension Tests of Thin Gage Metals and Light Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 24, Part II, p. 997 (1924).

Bend and twist tests were performed in the hand-operated machines, supplied by the W. & T. Avery Co.

Test Data:

A summary of the test data obtained on each sample coil of wire is presented in Table I. The information includes values for tensile strength, percentage elongation, twists and bends to failure, modulus of elasticity, endurance limit, and endurance ratio, that is, the ratio of endurance limit to tensile strength.

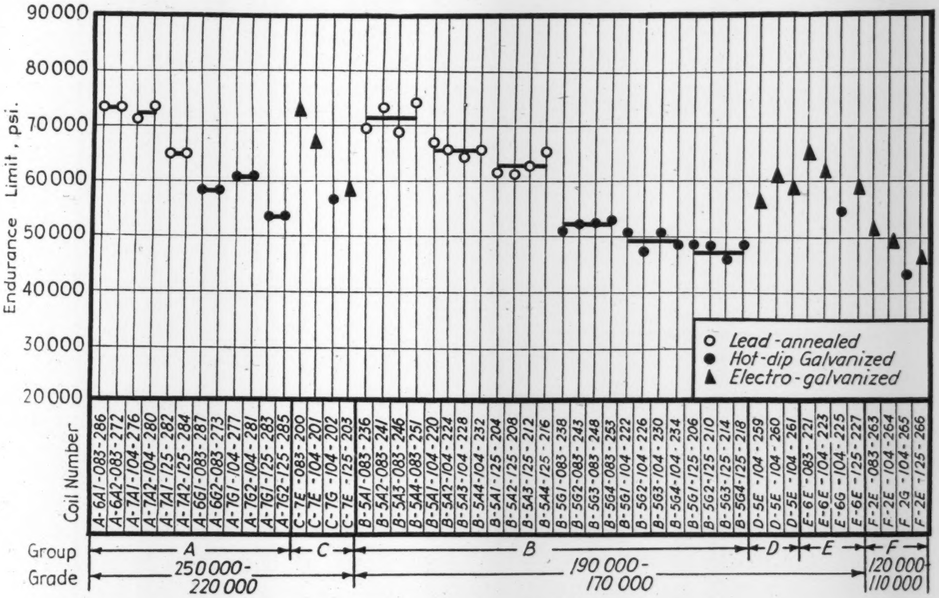

FIG. 5.—Endurance Limits of All Wires Tested.

Typical *S-N* curves on wires of different diameters from groups B and A, in the lead-annealed and hot-dip galvanized conditions, are shown in Figs. 3 and 4. It will be observed that on some of the curves there was a range of stress, varying from 1000 to 3000 psi. above the endurance limit, in which a number of the specimens broke at less than 10,000,000 cycles, while others, tested at the same stress, continued to run unbroken over the 10,000,000 cycle mark. It is thought that in this critical stress range, where the *S-N* curve is approaching the horizontal, a given degree of nonuniformity in the fatigue resistance of the specimens, such as that due to minute differences in surface condition, results in a greater variation in cycles to failure than at the higher stresses where the slope of the curve is steeper.

To facilitate ready comparison, the endurance limits of the wires are plotted in Fig. 5. Horizontal lines have been inserted to represent the average of the test values obtained on sample coils of wire having the same diameter, similarly processed and made from the same melt of steel.

A comparison of the results obtained on the lead-annealed and hot-dip galvanized wires in groups A and B reveals the deleterious effect of the hot-dip galvanizing process on the endurance limits of the uncoated steel wires. Galvanizing depressed the endurance limits of the lead-annealed wires 11,000 to 20,000 psi., the adverse effect of the coating being somewhat less for the wires in group A than for those in group B.

In Fig. 6, the endurance limits of all of the hot-dip galvanized wires are plotted against their respective tensile strengths. Although there is a considerable scatter in the points due to differences in chemical composition, reduction by drawing and processing treatments, the results indicate that the 220,000 to 250,000 psi. grade wires have somewhat higher endurance limits than the 170,000 to 190,000 psi. grade. The endurance limit of the one coil of 110,000 to 120,000 psi. grade was lower than those of the two harder grades. The improvement in the fatigue properties of the wires as the tensile strength increased was accompanied by a loss in ductility as revealed by a smaller percentage elongation at failure in the tension test.

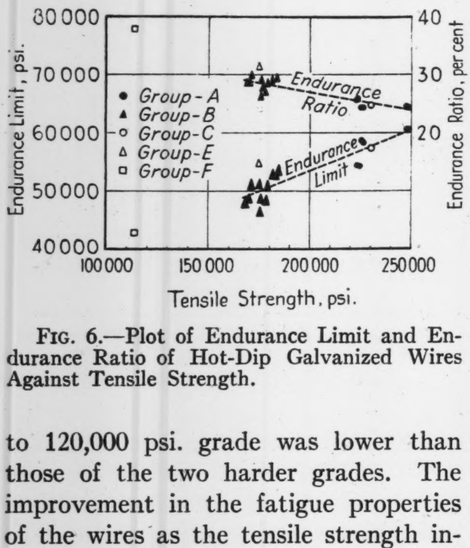

FIG. 6.—Plot of Endurance Limit and Endurance Ratio of Hot-Dip Galvanized Wires Against Tensile Strength.

The plot of the endurance ratios of all of the hot-dip galvanized wires against their respective tensile strengths, in Fig. 6, shows that this ratio becomes smaller as the tensile strength of the wire is increased. Reference to the data obtained on the lead-annealed and the electro-galvanized wires in Table I reveals a similar relationship between the endurance ratio and the tensile strength for wire in these conditions.

The effect of drawing on the endurance limit of the zinc-coated wires is illustrated by Fig. 7, in which the endurance limits of the wires, from each sample group, are plotted against the wire diameter. It will be observed that in every case some improvement was effected in the endurance limit as the reduction by drawing was increased. The break in the curve for the samples from group A is due to the use of a lower carbon steel for the 0.082-in. diameter wire than for the two larger sizes. A similar effect may be noted between the endurance limits of the lead-annealed wires of various diameters.

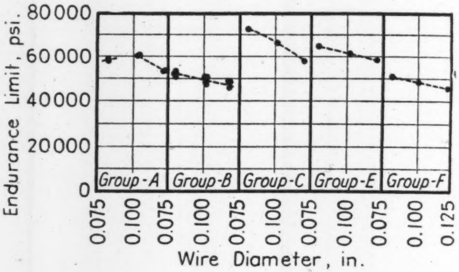

FIG. 7.—Endurance Limits of Zinc-Coated Wires from Each Group Plotted Against Wire Diameter.

It will be observed from the data in Table I that wires coated by electrolytic processes had endurance ratios slightly exceeding those obtained on the hot-dip galvanized wires of similar grade. Although variations in chemical composition, drawing, and heat treatment of the base wires may preclude a direct comparison of the relative effect of the two coatings, the results point to the superiority of the electrodeposited coatings over those applied by the hot-dip method, in so far as the fatigue resistance of the finished wires is concerned.

A number of tests were made to determine the effect of coating thickness on the endurance limit of hot-dip galvanized wire. Two coating thicknesses were employed, namely, those required to withstand four and three-and-one-half dips, respectively, in the Preece test. The samples were obtained by applying the coatings to wire from corresponding reels. The results of the tests, presented in Table II, indicate that within the range investigated coating thickness had no perceptible effect on the endurance limit of the wire.

TABLE II.—EFFECT OF COATING THICKNESS ON ENDURANCE LIMIT.

Coil Number	Weight of Zinc Coating, oz. per sq. ft.	Endurance Limit, psi.		Endurance Ratio, per cent	
		4 Dip Coating	3½ Dip Coating	4 Dip Coating	3½ Dip Coating
B-5G1-083-238	0.88	50 800	......	28.4	....
B-5G1-083-239	0.68	......	52 500	....	29.6
B-5G2 083-243	0.89	52 500	......	29.0	....
B-5G2-083-244	0.67	......	52 200	....	28.8
B-5G3-083-248	0.85	52 600	......	28.9	....
B-5G3-083-249	0.69	......	53 300	....	28.6
B-5G4-083-253	0.90	53 000	......	29.0	....
B-5G4-083-254	0.70	......	52 500	....	28.7

The average number of twists and bends to failure, along with the percentage average variation in the results obtained on the specimens from each coil, are shown in Table I. Plots of twists and bends to failure against the endurance limits of the wires revealed no definite correlation between these values.

Discussion of Test Results:

Several investigators have drawn attention to the detrimental effect of the hot-dip galvanizing process on the endurance limit of steel wire. In many of these researches, however, the comparison has been made between the endurance limit of the galvanized wire and that of the same wire chemically stripped of its coating or machined and polished to a smooth steel surface.[6, 4]

The injurious influence of the hot-galvanizing process on the fatigue properties of steel wire has been attributed both to surface notching caused by the etching action of the pickling solution and hot zinc on the wire surface and to the presence of the hard brittle zinc-iron alloy layers adjacent to the steel. Chemical stripping of the coated wire may remove the alloy layers, but it does not restore the surface of the steel to the condition prior to galvanizing. Similarly, when the coated wires are machined to the steel and polished, the surface condition is entirely changed and the endurance limit will probably exceed that of the wire before subjection to the coating process. A truer measure of the effect of the hot-galvanizing process on the fatigue properties of wire than is given by either of the above methods is to test the material before and after galvanizing as was done in this investigation.

The micrographs in Fig. 8 illustrating the surface condition of the steel in the zinc-coated wires, reveal the irregularities or notches that have always

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[6] S. M. Shelton and W. H. Swanger, "Fatigue Properties of Steel Wire," *Research Paper 754*, Nat. Bureau Standards (1935).

been associated with the relatively low endurance limits of these materials.

To investigate the influence of cyclic reversed stressing on the zinc coatings, microscopic examinations were made of longitudinal sections before and after subjection to 10,000,000 cycles of reversed stress at the endurance limit. The micrographs in Fig. 9 show that the hot-dip coatings during this treatment developed closely spaced hair cracks perpendicular to and terminating at the steel surface. Similar cracks could not be found in the electrodeposited coatings on specimens subjected to the same treatment. The absence of hair cracks in the electrogalvanized coatings after reversed stressing and the consequent elimination of a source of stress concentration which was active in the hot-dip coatings, may help to explain the higher endurance ratios obtained on the wires coated by the former process. Closely spaced hair cracks, of the type referred to above, could not be detected in the coating on specimens straightened in a tension testing machine, an observation which precluded the possibility that these cracks were produced in the straightening process.

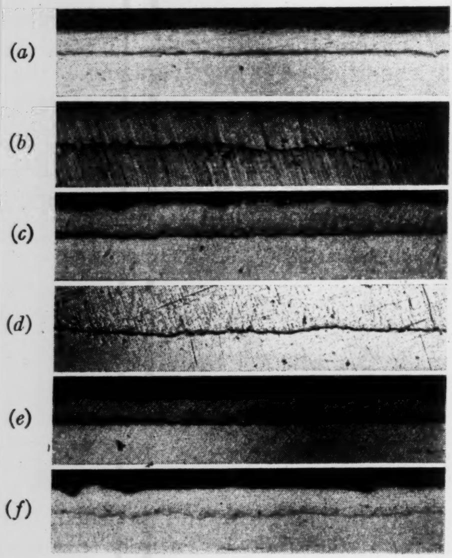


FIG. 8.—Contour of Parting Line Between Coating and Steel. Unetched (× 50).

(a) A-7G1-125-283
(b) B-5G2-125-210
(c) C-7E -104-201
(d) D-5E-104-260
(e) E-6E 104-223
(f) F-2E-104-264

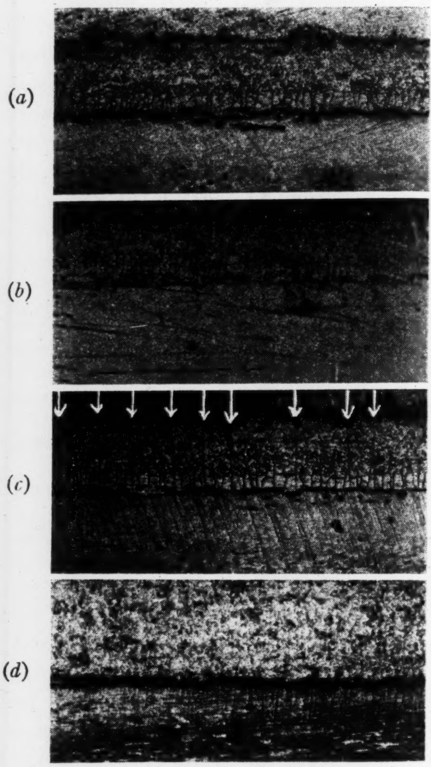


FIG. 9.—Hair Cracks Developed in Hot-Dip Coatings After Fatigue Stressing. Etched (× 100).

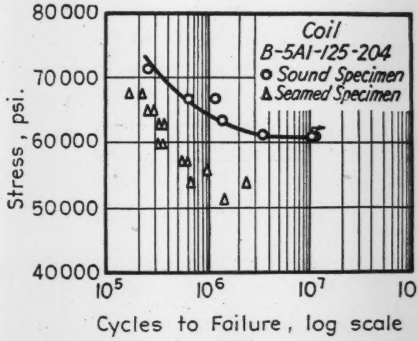
(a) As-received.
(b) Tensile straightened.
(c) After fatigue stressing B-5G2-125-210 hot-dip galvanized.
(d) After fatigue stressing D-5E -104-259 electrogalvanized.

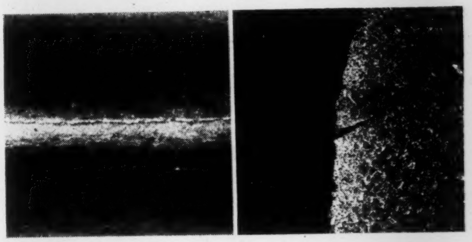
Macroscopic examination of the surface of the hot-dip coated fatigue specimens, after test, revealed the presence of circumferential corrugations or stress lines near the mid-span where the highest reversed bending stresses prevailed. Near the free ends of the specimens, where the stresses were relatively low, the appearance of the coated surface remained unchanged after test.

Hot-dip galvanizing depressed the endurance limits of the 170,000 to 190,000 psi. grade lead-annealed wires by 21 to 28.4 per cent. The corresponding reduction for the 220,000 to 250,000 psi. grade wire varied from 15.4 to 20.5 per cent. It might have been expected from the relative ductilities of the two materials that the endurance limits of the harder wires would have suffered the greater reduction in the galvanizing process. For the present the difference in the relative effect of the hot-dip coatings on the endurance limits of the two grades of wire remains unexplained.

From results obtained by other investigators in fatigue tests on steel wire, it may be shown that an increase in the tensile strength or carbon content of the material is generally accompanied by a decrease in the endurance ratio.[7, 8] The data presented here demonstrated that a similar relationship exists between the endurance limits of zinc-coated steel wires of various strengths, regardless of the method by which the coating is applied. The effect of the tensile strength or the carbon content of the wire on the endurance ratio has been partially attributed, by some authorities, to surface decarburization.[9] Traces of this condition were found in some of the sample groups but it was not common to all of the wires tested.

The utility of the fatigue test for detecting surface flaws on wire is illustrated by the results obtained on the lead-annealed coil No. B–5A1–125–204. The test values, plotted in Fig. 10, fall into two groups. Through one of these an *S-N* curve was drawn which agreed closely with those obtained on three coils of wire of a corresponding grade and diameter and drawn from the same melt of steel. The second group roughly defined a curve located considerably below the aforementioned one, all of the points on which indicated failure at less than 2,600,000 cycles of reversed stress. Subsequent examination of the specimens revealed that all of those which gave low and erratic values in the test contained one or more longitudinal surface seams similar to that illustrated in Fig. 10. The specimens which defined the normal fatigue curve showed





Surface (× 10). Section (× 50).

Seam in Wire.

FIG. 10.—Effect of Surface Flaw on Fatigue Test Results.

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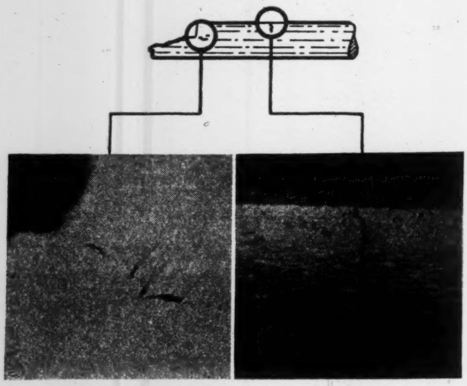
[7] A. V. de Forest and L. W. Hopkins, "The Testing of Rope Wire and Wire Rope," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 398 (1932).

[8] Discussion by H. J. Godfrey of paper by C. P. Wampler and N. J. Alleman, "Fatigue Tests of Wire," ASTM BULLETIN, No. 101, December, 1939, p. 18.

[9] E. T. Gill and R. Goodacre, "Some Aspects of the Fatigue Properties of Patented Steel Wires," *Journal*, Iron and Steel Inst., Vol. II, p. 293 (1934).

no evidence of this condition. Visual examination of the coil from which the above specimens were obtained revealed that surface seams occurred intermittently along a considerable portion of its length.

A few of the fatigue specimens broke with a split type of fracture. Microscopic study of these fractures invariably revealed the presence of elongated nonmetallic inclusions located near the shoulder of the break. A micrograph illustrating the condition in the vicinity of such a fracture is shown in Fig. 11. In this particular specimen a fatigue crack, in the process of development, was detected about ¼ in. from the point where failure occurred.



Unetched. Etched.

FIG. 11.—Nonmetallic Inclusion Located at Split Fatigue Fracture and a Second Fatigue Crack in the Process of Development (× 50).

Although the twist and bend test results could not be directly correlated with the endurance limits of the wires, the tests proved useful in that they furnished a convenient method of assessing the uniformity of material and detecting brittleness therein. The twist test results appeared to be strongly influenced by the surface condition and ductility of the outer fibers of the wire. Hot-galvanizing, in general, depressed the twists and bends to failure obtained on the lead-annealed wire, the effect being more marked in the wires in group B than in the harder wires in group A.

Special attention was directed to the type of fractures obtained in the twist and bend tests. Poor fractures of a split type generally occurred in specimens containing nonmetallic inclusions.

SUMMARY

The more significant points deduced from the test data presented in this paper include the following:

1. Within the limits of tensile strength investigated, namely, 110,000 to 256,000 psi., the endurance limits of the zinc-coated wires increased with the tensile strength while the endurance ratio diminished.

2. Hot-dip galvanizing depressed the endurance limit of the lead-annealed wire 15.4 to 28.4 per cent, depending to some extent on the grade.

3. No perceptible difference was observed between the endurance limits of the wire hot-galvanized with four and three-and-one-half dip coatings.

4. Reduction by drawing, within the range of wire sizes investigated, increased the endurance limits of the wires in both the lead-annealed and zinc-coated conditions.

5. Steel wires coated by electrolytic processes showed, in all three grades, somewhat higher endurance ratios than were obtained on wires coated by hot-dip methods.

6. Hot-dip coatings developed, under reversed stressing, closely spaced hair cracks perpendicular to and terminating at the surface of the steel. In the electrodeposited coatings no evidence of such cracks could be found after similar treatment.

7. Lack of uniformity in the test results and the abnormal fractures obtained on defective wires, containing surface seams or nonmetallic inclusions, demonstrated the utility of the fatigue test for detecting faulty materials.

8. Although no definite correlation could be observed between the bend and twist test results and the endurance limits of the wire, these tests proved useful in assessing the uniformity of the wire and detecting brittleness therein.

Acknowledgments:

The author is indebted to the Hydro-Electric Power Commission of Ontario for permitting publication of these data. He also wishes to acknowledge the assistance of E. J. Mason who prepared the micrographs.

DISCUSSION

Mr. H. J. Godfrey[1] (*presented in written form*).—The paper by Mr. Watt on the fatigue properties of zinc-coated steel wire is one of the first in which a true comparison has been made between the fatigue properties of plain and coated wire.

There are many factors affecting the fatigue properties of wire and it has been our experience to find decarburization as one of the most important factors. Although most of our tests have been made on plain wire in the as-drawn condition, we have recently made a few tests on wire with various types of zinc coatings. The results have shown that in cases where decarburization was present the effect of the zinc coating on the fatigue properties was entirely eliminated.

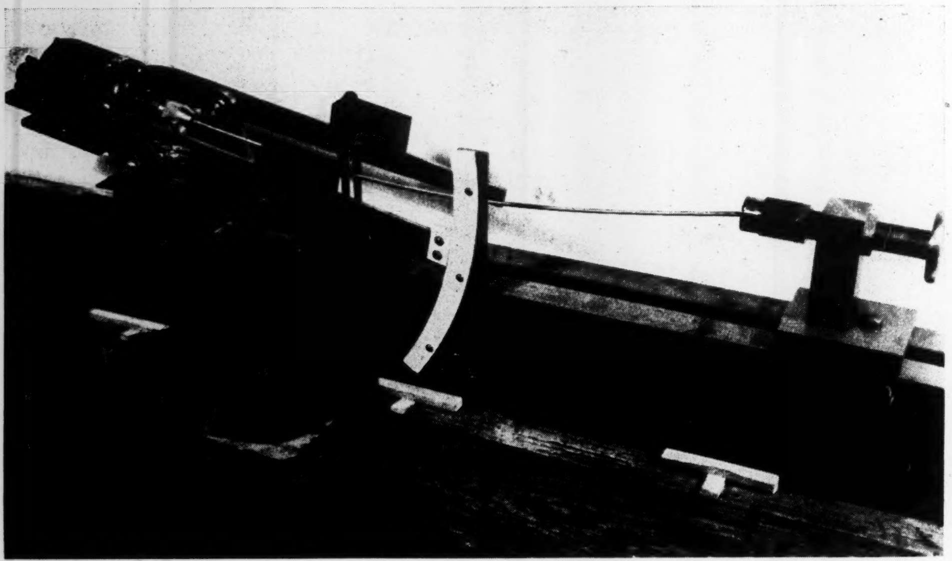


Fig. 1.—Fatigue Testing Machine.

For the purpose of carrying on these fatigue tests on wire we have developed a wire fatigue testing machine similar to that described by Mr. Watt. This machine is shown in the accompanying Fig. 1. The machine consists of a base on which a swinging frame is placed at one end and a tailstock at the other end. A motor is attached to the swinging frame and the wire specimen is held by a chuck fitted to the shaft of the motor and a thrust bearing in the tailstock. The wire is flexed to any desired amount by turning the tailstock, thus applying an end thrust to the specimen. In this manner the wire is stressed as a strut, the ends of which are not fixed. Simultaneously the motor rotates the wire on its own axis causing a complete reversal of the bending stress for each revolution.

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[1] Engineer of Tests, Fritz Engineering Laboratory, Department of Civil Engineering, Lehigh University, Bethlehem, Pa.

The wire is bent in a vertical plane and the angle of inclination is measured by a scale reading to 0.05 deg. When the specimen fractures, the swinging frame is thrown out of balance and a mercury switch automatically stops the motor. Specimens as large as $\frac{1}{4}$ in. in diameter have been tested satisfactorily.

MR. W. H. SWANGER.[2]—I was pleased to see that Mr. Watt's results agreed quite well with published results of fatigue tests on coated wire, in which the effect of the coating itself was evaluated by making tests of coated wire and then removing the coating from other portions of the wire and making tests on the stripped material. In this procedure there was always a question as to how much of the difference in fatigue limits for the two kinds of wire was caused by the changes in the surface of the steel resulting from putting on the coating and then taking it off. Mr. Watt has been able to take wire up to the point where it was ready for coating, get his samples for fatigue tests and then get samples of the same wire, after coating, for similar tests. It is interesting that Mr. Watt's results for the damaging effect of a hot-dip coating on a high-strength steel wire are in close agreement with previously published results obtained with stripped wire.

MR. D. G. WATT[3] (*Author's closure*).—Mr. Godfrey's tests have shown that application of zinc coatings to decarburized wire does not depress the endurance limit of the uncoated wire. Where definite decarburization is present the endurance limit of the wire in either the coated or uncoated condition will be very low, the fatigue properties in reversed bending being mainly those of the soft ferrite surface.

Metallographic study of the materials used in our investigation showed some evidence of decarburization in the 0.60 to 0.75 per cent carbon wires in group A but little, if any, in the 0.50 per cent carbon wires in group B. In the light of Mr. Godfrey's tests it would be interesting to know whether the difference in the endurance limits of the uncoated and coated wire becomes smaller as the degree of decarburization becomes more pronounced. If this were so it might offer an explanation for the fact that hot galvanizing depressed the fatigue limits of the uncoated wires in group B 21 to 28 per cent, while the corresponding reduction for the wires in group A was only 15.4 to 20.5 per cent.

The fatigue testing machine used in Mr. Godfrey's experiments is similar to the Haigh-Robertson type with the exception that the swinging headstock is pivoted horizontally instead of vertically, a feature which simplifies considerably the mechanical construction of the machine. In Mr. Godfrey's machine the wire is flexed in the vertical rather than the horizontal plane and accordingly the dead weight of the specimen will introduce a small error in the stress as calculated by the Euler column formula from the angle of inclination at the free ends of the specimen. This error is so small, however, that it may well be neglected.

I am glad that Mr. Swanger has emphasized the distinction between our method of comparing the endurance limits of the wire before and after the galvanizing process, and results published elsewhere in which the comparison was made between the endurance limit of galvanized wire and that of the same wire chemically stripped of its coating. Both methods of comparison show the detrimental effect of the hot-dip coating on the fatigue properties of the wire. However, as shown below, the spread between the endurance limits of steel wire, 0.122 in. in diameter, before and after galvanizing is slightly greater than that between the endurance limits of the chemically stripped and galvanized wire.

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[2] Metallurgist, National Bureau of Standards, Washington, D. C.

[3] Assistant Testing Engineer, The Hydro-Electric Power Commission of Ontario, Toronto, Ont., Canada.

	Endurance Limit, psi.
Before galvanizing	62 300
Galvanized	51 300
Chemically stripped of coating	60 200

FATIGUE STRENGTH OF 2-IN. DIAMETER AXLES WITH SURFACES METAL COATED AND FLAME HARDENED

By O. J. Horger[1] and T. V. Buckwalter[1]

Synopsis

Fatigue investigation was made of axles tested as rotating cantilever beams 2 in. in diameter. Axles with and without stress concentration were sprayed with a metal coating. Also axles with stress concentration were locally flame hardened Fatigue tests were made on these specimens and comparison made with similar axles without surface treatment.

The base axle materials were S.A.E. 1035 and 1045 steels and the sprayed metal was 0.40 and 1.20 per cent carbon wire stock. The nature of the stress concentration was a press-fitted wheel. Test results with plain specimens indicated little difference in endurance limit of coated axles over that for uncoated axles. Press-fitted wheel and axle assemblies showed a great increase in endurance limit of the coated and flame-hardened axles over the untreated axles.

A project on axle research was initiated about 5 yr. ago and various progress reports have been periodically published. The laboratory tests reported here represent one phase of this general program and presents a study of the fatigue strength of axles.

Various investigators[2, 3, 4] have reported how the presence of press-fitted gears, wheels, bearings and similar members develops locally high stresses in the axle near the edge of the fitted member to produce axle fatigue failure at this location. This clamping action superimposed on the usual bending stresses in the axle results in an extremely low endurance limit which is only 20 to 50 per cent of that for the same axle steel without the press fit. This paper submits the results of laboratory tests indicating the improvement in axle fatigue strength which was obtained by certain methods of surface treatment of the axle in the region of the press-fitted member. The two methods of surface treatment discussed here are metal spraying and flame hardening.

Material

All axles tested with press-fitted wheels were S.A.E. 1045 steel, normalized and tempered. Some plain specimens were also tested which were essentially S.A.E. 1035 steel, annealed. Table I gives the chemical analysis and physicial properties determined by test.

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[1] In Charge of Railway Engineering and Research, and Vice-President, respectively, The Timken Roller Bearing Co., Canton, Ohio.

[2] A. Thum and F. Wunderlich, "Einfluss von Einspann- und Kraftangriffstellen auf die Dauerhaltbarkeit der Konstruktionen," *Zeitschrift* des Verenies deutscher Ingenieure, Vol. 77, No. 31, August 5, 1933, p. 851.

[3] R. E. Peterson and A. M. Wahl, "Fatigue of Shafts at Fitted Members with a Related Photoelastic Analysis," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 57, p. A-1 (1935).

[4] T. V. Buckwalter and O. J. Horger, "Investigation of Fatigue Strength of Axles, Press-Fits, Surface, Rolling and Effect of Size," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 25, p. 229 (1937).

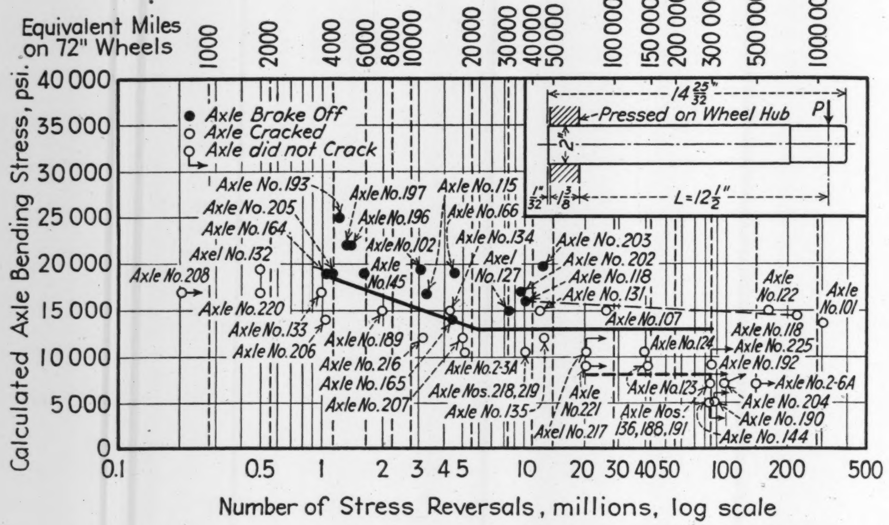

FIG. 1.—*S-N* Data on Press-Fitted Axles Without Surface Treatment of Wheel Seat.

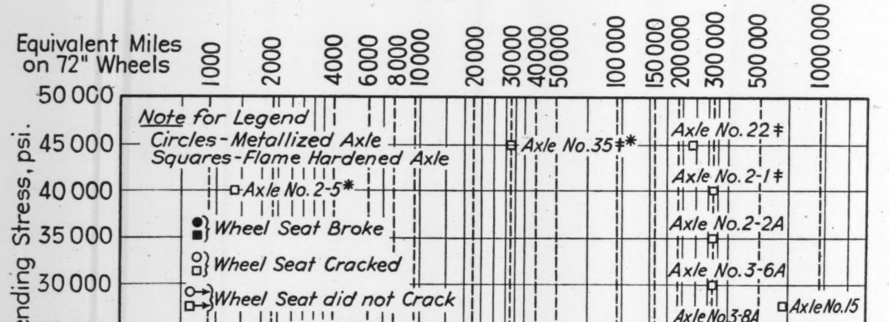

FIG. 2.—*S-N* Data on Press-Fitted Axles Having Metal Coated and Flame-Hardened Wheel Seats.

The chemical analysis for the sprayed layer indicated in Table I was obtained from a sample of the actual deposit of material on the axle. Typical photomicrographs of these materials are shown in Figs. 7 and 8.

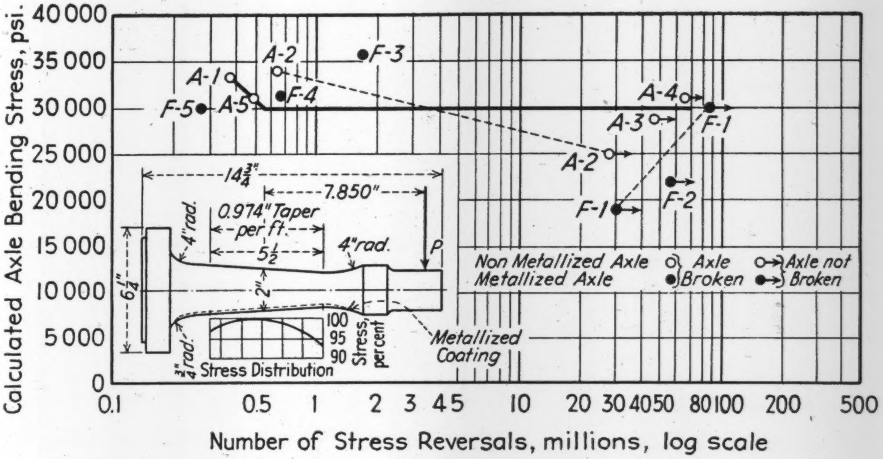

FIG. 3.—*S-N* Data on Plain Specimens with and Without Metal Coating.

TABLE I.—CHEMICAL ANALYSIS AND PHYSICAL PROPERTIES.

	Axles (S.A E. 1045) with Press-Fitted Wheels		Plain Specimens S.A.E. 1035	
	Axle	Coating After Spraying[b]	Axle	Coating After Spraying
Carbon, per cent	0.45 to 0.49	1.16	0.29 to 0.35	0.56
Manganese, per cent	0.67 to 0.77	0.34	0.43 to 0.58	0.40
Silicon, per cent	0.20 to 0.26	0.10	0.12 to 0.24	0.15
Phosphorus, per cent	0.013 to 0.017	0.022	0.013 to 0.016	0.013
Sulfur, per cent	0.031 to 0.037	0.024	0.031 to 0.032	0.029
Tensile strength, psi.	88 800	10 000 to 13 000	....	6 500 to 16 900[c]
Yield strength, psi.[a]	47 800	....	....	....
Elongation in 2 in., per cent	32	....	....	....
Reduction of area, per cent	48.5	....	....	....
Brinell hardness	163 to 197	[b]	156 to 201	235 to 269
Endurance limit 2 in. plain specimen rotating cantilever beam	34 400	....	....	....
Heat treatment (before metallizing or flame hardening)	Normalized 1620 F. Tempered 1115 F.		Annealed	

[a] Determined by "drop of the beam" method.

[b] Furnished by Metallizing Engineering Co. where hardness of sprayed layer was Rockwell C 30 to 32 or Brinell (1000-kg. load) 260.

[c] Furnished by T. S. Fuller, General Electric Co. for standard tension specimen 0.505 in. in diameter made of sprayed metal as well as ultimate compressive strength values using cylinders 1 in. high by ½ in. in diameter which gave 87,500 and 91,000 psi.

The chemical analysis of the wheels used as press-fitted members on the S.A.E. 1045 axles were in accordance with A.A.R. Specification M-107-36[5] or A.S.T.M. Specifications A 57 - 39[6] for multiple-wear wrought steel wheels. The Brinell hardness of the wheels used measured 250 with 110,000 psi. ultimate strength and 60,000 psi. yield strength as obtained by "drop of the beam." Wheels were generally fitted to the axle 0.003 to 0.004 in. tight except where noted.

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[5] Manual of Standard and Recommended Practice, Assn. Am. Railroads, Mechanical Division, Chicago (1936).

[6] Standard Specifications for Multiple-Wear Wrought Steel Wheels (A 57 - 39), 1939 Book of A.S.T.M. Standards, Part I, p. 201.

All specimens tested had a nominal diameter of 2 in. The design of axles tested is shown in Figs. 1, 2 and 3.

Location of the flame-hardened region of the axle under the wheel fit is indicated in Fig. 2. The setup for flame hardening[7] consisted of a W-23 blowpipe and a flame head for heating the specimens. The flame head contained three rows of seven No. 56 drill-size tips per row. The flame-hardening head was mounted symmetrically with the longitudinal axis of the shaft and adjusted so that the flames just cleared the shaft during the heating cycle. An allowance of ¼ in. between the last flame and the extreme end of the shaft was made to prevent an increased depth of hardened material at this point.

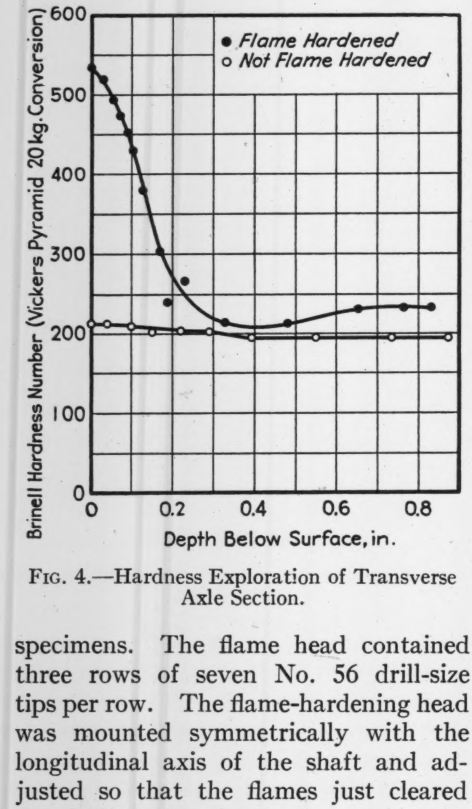

FIG. 4.—Hardness Exploration of Transverse Axle Section.

Quenching of the material was obtained by using pressure water spray quenches mounted over and under the specimen and controlled by a quick-acting gate valve. The axles were treated as follows:

1. Heat for 35 sec. while spinning in a lathe at 150 r.p.m.

2. Quench specimen simultaneously with gas shut off.

3. Draw immediately in oil for 2 hr. at 400 F.

The flame-hardened region was ground to obtain the proper wheel fit. Hardness exploration on transverse axle sections is shown in Fig. 4. Photomicrographs taken near the surface and in the core are given in Fig. 8 (*a*) and (*b*).

Location of the sprayed metal layer in the region of the wheel fit is shown in Fig. 2. A screw type of thread 0.025 in. deep was turned on this portion of the axle before spraying with Sprasteel 120.[8] Thickness of the sprayed layer was $\frac{1}{32}$ in. over the tops of the threads and this surface was ground before fitting the wheel so as to leave $\frac{1}{64}$ in. thick layer as tested.

Design of the plain specimens of S.A.E. 1035 steel with and without the metal sprayed layer is shown in Fig. 3.[9] The metallized specimens were turned to 1¾ in. in diameter as shown, sand blasted, sprayed with 0.40 per cent carbon wire stock and then machined and polished. The final diameter was 2 in. which left ⅛ in. thick layer of sprayed metal on the specimens as tested.

TEST CONDITIONS

All axles were tested as rotating cantilever beams in machines previously de-

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[7] Flame hardening done by Linde Air Products Co.

[8] These tests were made some time ago and the type of threaded surface used here is not recommended by Metallizing Engineering Co. Instead of semicircular groove or thread with the tops roughened and ridged over is proposed. This change of thread shape is to reduce stress concentration at the bottom of the thread.

[9] This group of specimens furnished ready for test by T. S. Fuller of General Electric Co.

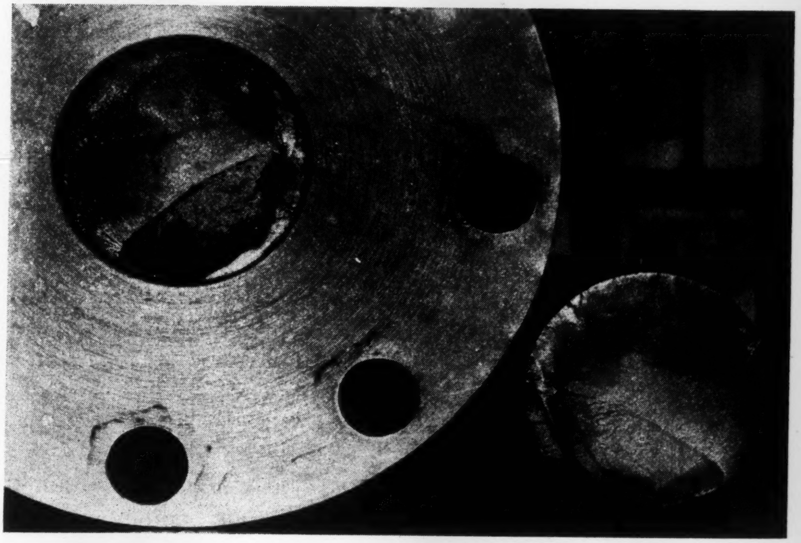

FIG. 5.—Typical Fatigue Failure of Press-Fitted Axle Assembly.

FIG. 6.—Fatigue Fracture of Metal-Coated Specimen No. F-3.

scribed.[10] Speed of these machines varied from 1400 to 2100 r.p.m. Endurance limit values are based on 84 million stress reversals for press-fitted axles and about 50 million for plain specimens.

Stresses mentioned in this paper are nominal bending stresses in the axle based on the usual beam formula where stress equals the bending moment divided by the section modulus. For the case of the axles with the press-fitted

TABLE II.—FATIGUE DATA ON PRESS-FIT AXLE TESTS.

Axle	Wheel Fit on Diameter, in.	Axle Bending Stress, psi.	Revolutions in Millions[g]	Remarks
AXLES METAL COATED IN WHEEL FIT				
No. 231	0.0056	25 000	1.92	Broke in wheel fit
No. 232	0.0073	22 000	2.30	Broke in wheel fit
No. 228	0.0053	22 000	168.6	Not cracked
No. 230	0.0057	21 000	84.3	Not cracked
No. 229	0.0060	20 000	84.9	Not cracked
No. 180[a]	0.0032	19 000	115.1	Not cracked
		22 000	66.7	Broke in wheel fit
No. 181[a, b]	0.0030	19 000	115.1	Not cracked
		22 000	7.33	Broke in wheel fit
AXLES FLAME HARDENED IN WHEEL FIT				
No. 35[c]	0.0063	45 000	8.64	Cracked in wheel fit
No. 22[d]	0.0056	45 000	68.1	Cracked in wheel fit
No. 2 5[c]	0.0051	40 000	0.38	Cracked in wheel fit
No. 2-1[d]	0.0061	40 000	84.3	Cracked in wheel fit
No. 2 2A[e]	0.0046	35 000	84.3	Cracked in wheel fit
No. 3 6A	0.0052	30 000	84.3	Cracked in wheel fit
No. 15[f]	0.0050	28 000	191.0	Cracked in wheel fit
No. 3 8A	0.003	25 000	85.1	Not cracked
No. 30	0.0054	25 000	172.3	Not cracked
No. 2-4A	0.0055	19 000	84.3	Not cracked

[a] Surface of this axle sand blasted instead of having threaded surface previous to metallizing.

[b] Stainless steel sprayed on this axle instead of Sprasteel.

[c] Axle broke off in body outside of wheel fit.

[d] Axle rolled in body.

[e] Maximum crack depth, 0.015 in.

[f] Maximum crack depth, 0.022 in.

[g] Equivalent revolutions on 72-in. diameter wheel obtained by dividing 281.

TABLE III.—FATIGUE DATA ON PLAIN SPECIMEN TESTS.

Axle	Bending Stress, psi.[a]	Revolutions in Millions	Remarks
SPECIMENS NOT METALLIZED			
A-1	33 300	0.378	Broke up fillet 2 deg.
A-5	31 000	0.496	Broke base fillet
A-4	31 000	66 428	Did not break
A-3	28 700	46 320	Did not break
A-2	25 000	28.539	Did not break
	32 800	0.630	Broke up fillet 1 deg.
SPECIMENS METALLIZED			
F-3	35 400	1.686	Broke base fillet
F-4	31 300	0.657	Broke at end coated Layer
F-5	30 000	0.279	Broke up fillet 3 deg. 30 min.
F-2	22 000	58.212	Did not break
F-1	19 000	30.060	Did not break
	30 000	89.450	Did not break

[a] When failure occurs in fillet the stress is given on basis of actual diameter at base of fillet. Where failure does not occur stress is given at center of taper section.

TABLE IV.—SUMMARY OF ENDURANCE LIMIT VALUES.

PRESS-FITTED AXLES (FIGS. 1 AND 2) OF S.A.E. 1045 STEEL

Surface Treatment	Endurance Limit in Wheel Seat to Prevent					
	Initiation of Fatigue Cracks			Breaking off of Axle		
	psi.	per cent	Stress concentration factor[b]	psi.	per cent	Stress concentration factor[b]
Normalized and tempered	8 000[a]	100	4.30	13 000	100	2.65
Metallized	21 000	262	1.64	21 000	162	1.64
Flame hardened	25 000	313	1.38	>45 000	>321	....

PLAIN SPECIMENS (FIG. 3) OF S.A.E. 1035 STEEL

Surface Treatment	Endurance Limit of Plain Section	
	psi.	per cent
Annealed	~30 000	100
Metallized	~28 000	93

[a] This value was previously published by in error as being 6000 psi.

[b] This value obtained by dividing the endurance limit of 34,400 psi. (Table I) for a plain specimen by that given for the press-fitted axle.

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[10] O. J. Horger and H. R. Neifert, "Fatigue Strength of Machined Forgings 6 to 7 in. in Diameter," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 723 (1939).

wheels the axle bending stress is given at the hub face and for the plain specimens in the tapered section at the plane of failure.

TEST RESULTS

Results of these various fatigue tests are given in Tables II and III and the values plotted in the S-N curves of Figs. 1, 2 and 3. Typical fractures are shown in Figs. 5 and 6. The endurance limit values for the different specimens are summarized in Table IV.

DISCUSSION

Press-Fitted Axles Without Surface Treatment:

Double values for the endurance limit are given in Table IV and Fig. 1 for the case of the axle with the press-fitted wheel. The lower value of 8000 psi. means that no fatigue cracks will develop in the axle wheel fit up to and including that stress while the higher value indicates that the axle will break off in the wheel fit above 13,000 psi. Between the lower and upper endurance limit values fatigue cracks develop in the wheel fit but do not appear to propagate to a depth greater than about $\frac{1}{8}$ in. which is not sufficient to cause the axle to actually break off unless the stress exceeds 13,000 psi.[11]

This phenomena of double endurance limit values does not exist for specimens without clamped or fitted members. Its existence here is believed to be a result of the repeated minute sliding action of the end face of the hub on the axle surface due to the alternate elongation and contraction of the axle fibers produced by variable bending stresses. Results of this action are noticeable by the corrosion and pitting which occurs in irregular areas accompanied by oxide debris near the ends of the press-fit. This sliding results in molecular attrition or mechanical abrasion[12] which in the presence of axle bending stresses causes the fatigue cracks to develop at very low stresses.

The stress concentration introduced by a fatigue crack once developed through such molecular attrition in the wheel fit of an axle is not as great as the weakening effect of molecular attrition itself. This condition is given in terms of stress concentration factors or over-all weakening effect due to the fitted member in Table IV. Some explanation for the fact that these incipient cracks will not progress to a point where the axle will break off unless the stress is increased above the high endurance limit value in Table IV may be found in tests by Moore.[13] He found that incipient fatigue cracks which had already developed in plain rotating beam specimens cut from railroad axles did not propagate until the bending stresses reached a value of about 50 per cent of the endurance limit of the virgin material.

Double endurance limit characteristics are not a result of laboratory test conditions but there is considerable proof of its existence from applications in service. Incipient fatigue cracks appear very early in axle life due to stress reversals of a low magnitude but in comparatively few cases do we find that the axles break off in the wheel fit. The propagation of these fatigue cracks to dangerous depths depends largely upon the number of occasional high stresses im-

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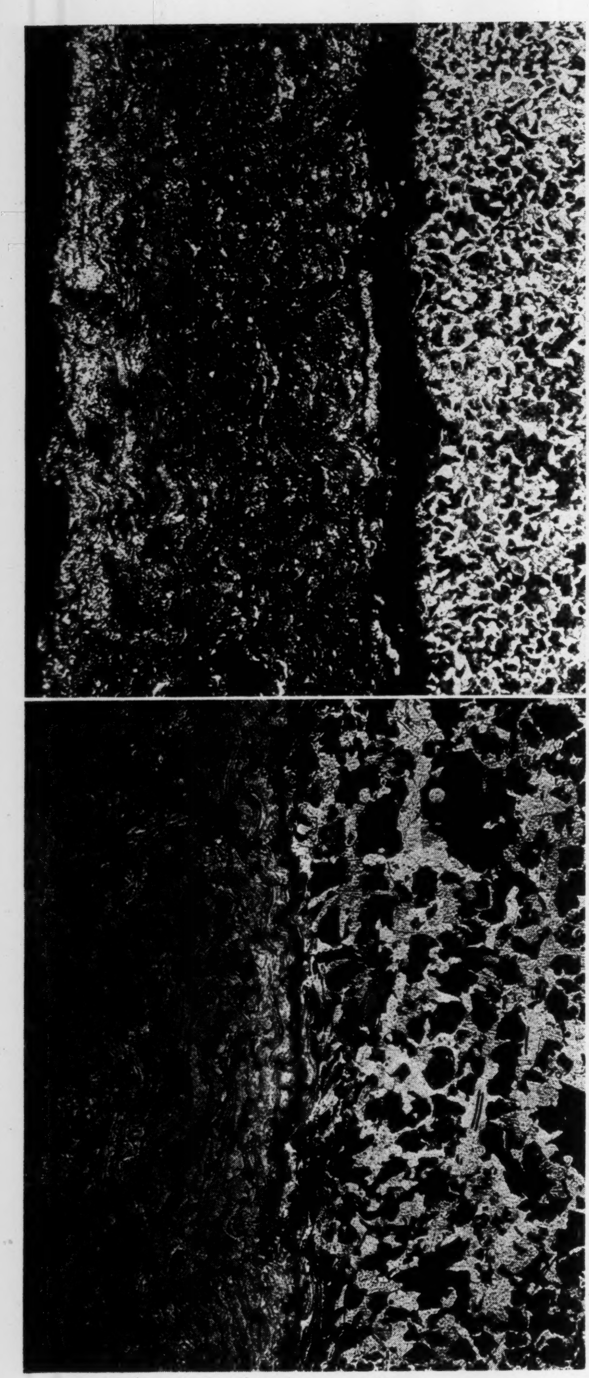
[11] Reason for double endurance limit values discussed by T. V. Buckwalter, O. J. Horger and W. C. Sanders, "Locomotive Axle Testing," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 60, p. 335 (1938). Contrary to views expressed then, later unpublished tests indicate that two endurance values exist independent of whether or not a bending moment is transmitted through the pressed-on member.

[12] G. A. Tomlinson, P. L. Thorpe and H. J. Gough, "An Investigation of the Fretting Corrosion of Closely Fitting Surfaces," *Journal and Proceedings*, Inst. Mechanical Engrs., Vol. 141, No. 3, pp. 223-249 (1939).

[13] H. F. Moore, "A Study of Fatigue Cracks in Car Axles," *Bulletin No. 165*, Engineering Experiment Station, University of Illinois (1927).



(*a*) Plain specimen without press fit No. F-3

(*b*) Axle with press-fitted wheel No. 180

FIG. 7.—Photomicrographic Studies of Transverse Sections of Metal-Coated Specimens. Upper layer is metal coating and lower is base material (× 100).

posed on the axle, that is, stresses which would be above 13,000 psi. Equipment is often subjected to disassembly for magnaflux inspection similar to the practice followed in the railroad and airplane industry. In such case the lower endurance limit value in Table IV becomes very important because the piece is scrapped if cracks appear. Where no such periodical examination is made of equipment, then the breaking off strength indicated by the higher endurance limit value becomes important. In any event it is desired to have both endurance limit values as large as possible or/and have a large spread between the two values.

Metallized Specimens:

A mechanical bond exists between the sprayed metal coating and the base material. It is intended that the roughened surface of the base material form keys or openings for the sprayed metal to penetrate. Doubt existed in some quarters as to whether the sprayed metal coating would loosen during the testing or in the mounting of the press-fitted wheels. The only time that separation of the sprayed layer occurred from the base was when specimens broke off above the endurance limit value, and Fig. 6 shows such a typical condition.

The number of specimens and stress levels at which the fatigue tests were made on the plain specimens with and without metallizing, Table III and Fig. 3, were not as conclusive as to the exact endurance limit value as would be desired. Also the failures in the 4-in. fillet leaves some question as to the influence of stress concentration. Inasmuch as some specimens were tested at a certain stress without failure followed by testing at an increased stress introduces the effect of prestressing. For these reasons the endurance limit values assigned in Table IV may be subject to some small corrections but they are interpreted as showing little or no reduction in endurance limit of the metal-coated axle over that for the base material. The photomicrograph in Fig. 7 (*a*) shows the region including the sprayed layer and the base material.

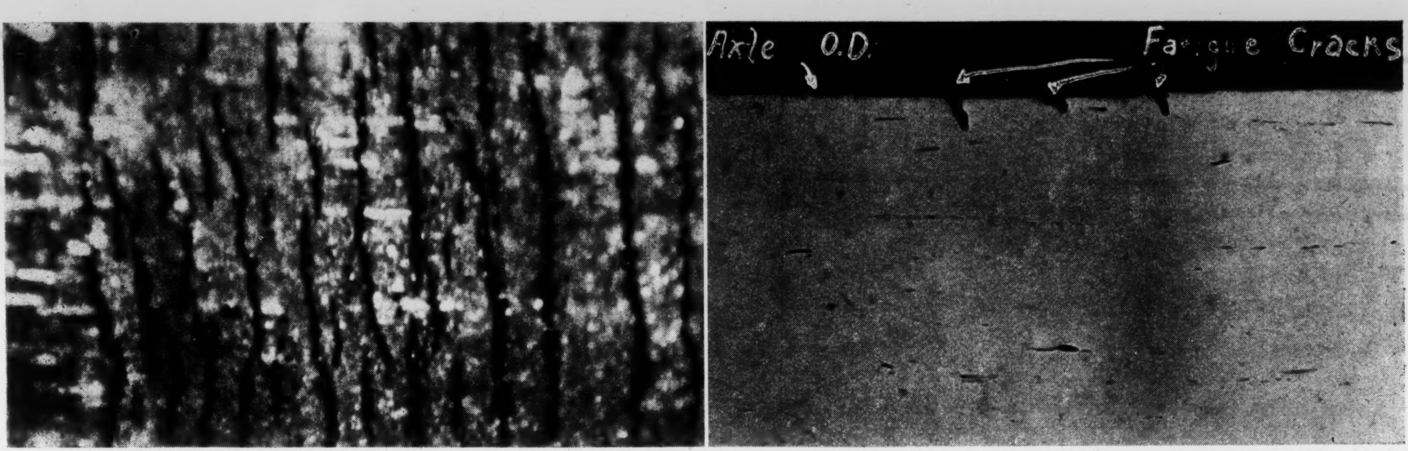
Results of fatigue tests made on press-fitted axles as outlined in Table II and Fig. 2 permit one endurance limit value of 21,000 psi. to be established for the case of the metallized axle at the wheel fit. In comparison with the untreated axle, Fig. 1 and Table IV, the metal coated axle has 2.6 times as great allowable cracking strength and 1.6 times as great allowable breaking off strength. Structural conditions existing in the sprayed layer and base material are indicated in photomicrograph of Fig. 7 (*a*).

There are many applications of machine parts where fitted members are pressed apart and cannot be reassembled because of loss of fit. The sprayed coating offers a means of reclaiming these parts by building up the surface and at the same time obtained a higher endurance limit than existed without the metal coating.

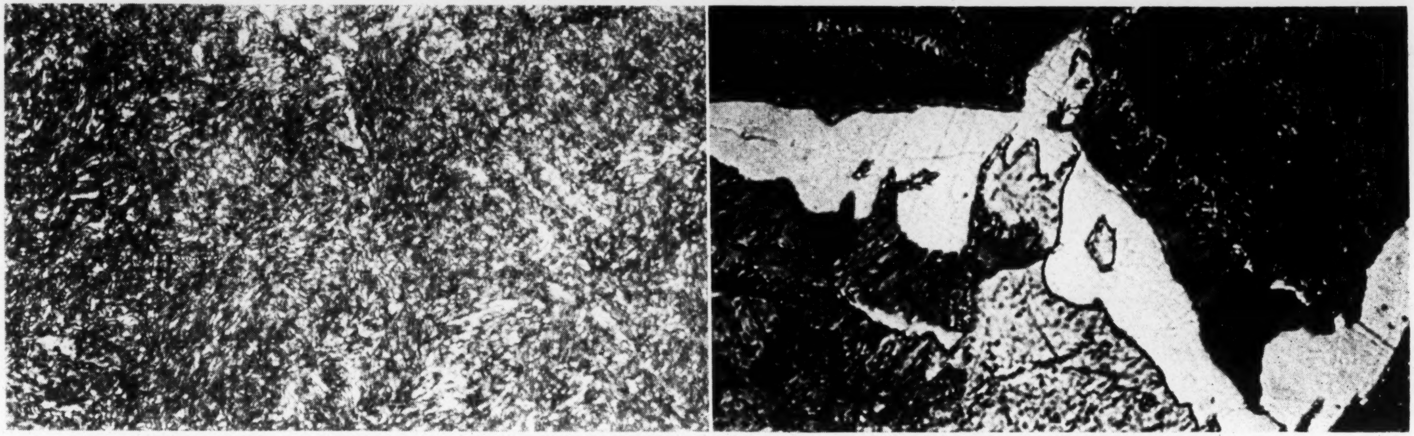
Flame-Hardened Axles:

Results of these tests given in Table II and Fig. 2 show that the endurance limit for cracking is 25,000 psi. while that for breaking is greater than 45,000 psi. or higher than that for an axle without a press-fitted wheel.

In order to test the axle at bending stresses in the wheel fit of 40,000 and 45,000 psi. it was necessary to roll the body of the axle so that it would not break off in the body. Typical fatigue cracks occurring in the wheel fit are evident from Fig. 8 (*a*) where a series of circumferential cracks develop within the fit near the hub face. Such a series of cracks is characteristic of axles which



(*a*) Series of circumferential fatigue cracks in axle wheel fit No. 2-1 (× 75). (*b*) Unetched longitudinal section of wheel seat showing fatigue cracks No. 3-6A (× 100).



(*c*) Structure near axle surface No. 3-8A (× 1000). (*d*) Core structure of axle No. 3-8A (× 1000).

Fig. 8—Photomicrographic Studies of Flame-Hardened Axles with Press-Fitted Wheels.

are strengthened by some means at the wheel seat and subjected to relatively high stresses. These cracks being very shallow, Fig. 8 (*b*), possibly indicate that, once one group of circumferential cracks are formed, the wheel fit is lost for the distance from the crack to the hub face. Then the next group of circumferential cracks develops and so on until about one-third to one-half the wheel fit length constitutes a mass of cracks. At this time the fit is no longer sufficient to hold the axle and wheel together and the wheel moves on the axle.

Endurance limit for a flame-hardened plain specimen 2 in. in diameter is not known. This accounts for the possible stress concentration factor less than 1 in Table IV where comparison is made with the endurance limit of the untreated plain specimen. Zimmerman[14] found an endurance limit of 52,000 psi. for a $\frac{7}{16}$-in. diameter square-notched flame-hardened specimen of the same material reported in this paper. This indicates a very high endurance limit for a plain specimen flame hardened, possibly better than 100,000 psi., and offers a partial explanation for the fact that the fatigue cracks in the wheel-fit tests were only very shallow at bending stresses up to 45,000 psi.

CONCLUSION

The use of the metal-coating and flame-hardening processes is not new but no data have been published concerning the fatigue properties of axles surface treated in this manner at the wheel fit.

Plain specimens 2 in. in diameter of annealed S.A.E. 1035 steel showed little change in endurance limit with and without the metal-coated layer.

S.A.E. 1045 steel axles with press-fitted wheels and metal coated in the wheel fit gave 1.6 to 2.6 times the endurance limit at the wheel fit portion of similar axle assemblies not metal coated.

S.A.E. 1045 steel axles with press-fitted wheels and flame hardened in the wheel fit gave about 3 times the endurance limit at the wheel-fit portion of similar axle assemblies not flame hardened.

Acknowledgment:

Appreciation is expressed here for the assistance of H. R. Neifert of the Timken Research Laboratory in making these fatigue tests. Also to Linde Air Products Co., Metallizing Engineering Co., and T. S. Fuller of the General Electric Co. for their cooperation in these tests.

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[14] J. H. Zimmerman, "Flame Treating," *The Welding Journal*, February, 1940.

DISCUSSION

MR. T. S. FULLER[1] (*presented in written form*).—The paper by Horger and Buckwalter has very appropriately pointed out that "Plain specimens 2 in. in diameter of annealed S.A.E. 1035 steel showed little change in endurance limit with and without the metal-coated layer."

I am able to offer evidence to substantiate this statement, but I cannot submit an explanation which satisfactorily accounts for the observed facts. Four sets of S.A.E. 1035 rotating cantilever-beam type of fatigue specimens, having the maximum stress in a section whose diameter was 0.4687 in. were prepared. Instead of undercutting the section of these smaller specimens, it was built up by metal spraying with steel of the same composition, in amount equal to one-eighth of the diameter. The increased diameter was considered when stress calculations were being made.

Sprayed metal adheres only to roughened surfaces. Before coating, therefore, the surfaces of one set of specimens were shot-blasted and of another set rough machined, the grooves or threads remaining after machining being 0.003 in. in depth.

In addition to endurance tests on the base metal and on the two sets of specimens described above, in order to show the notch sensitivity of the base material, tests were made on a fourth set prepared by notching. In each of the notched specimens, a V notch 0.035 in. deep having an included angle of 67 deg. and a radius at the bottom of 0.001 in. was cut circumferentially at the section of maximum stress. Stress calculations were based on the minimum diameter at the base of the notch.

The results follow:

Specimen	Endurance Limit, psi. (10,000,000 cycles)
Base metal	39 000
Shot blasted and metallized	34 000
Rough machined and metallized	31 500
Notched specimens	19 000

MR. J. M. LESSELLS.[2]—I wonder whether the authors could give us an explanation of the increased endurance limit as found by the spraying process. In other words, what influence does the surface finish have in a press-fit? Gough has given us a new term called Frettage Corrosion and a number of fatigue failures can be traced to this phenomenon. Possibly the increased endurance limit can be explained on the basis of the difference in contact between the sprayed metal and the ordinary metal in the press-fit.

MR. H. V. INSKEEP.[3]—I should like to ask whether any relationship has been found between the thickness of the flame-hardened case and the fatigue resistance? The reason I ask this question is that it may not be necessary to put on as thick a case as that shown. It is an economic advantage to make a case thinner, and it can easily be done. The results of these tests show great promise and certainly cost will be a factor in the adoption of the method.

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[1] Engineer of Materials, Works Laboratory, General Electric Co., Schenectady Works, Schenectady, N. Y.

[2] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[3] Development Engineer, The Linde Air Products Co., Newark, N. J.

MR. R. E. PETERSON[4] (*by letter*).—The results reported by Messrs. Horger and Buckwalter for flame-hardened axle seats are quite remarkable, better than a three-fold improvement being obtained. In this connection, it would be worth considering the application of induction hardening which possesses some advantages over flame-hardening; particularly if a considerable number of axles are to be hardened. The surface hardening technique would be preferable to rolling where the contour is irregular. If further improvement beyond that given by surface hardening the entire axle is desired, the grooved hub construction could also be used. The authors are to be congratulated on making a major contribution to the solution of the press fit problem.

MR. O. J. HORGER[5] (*author's closure*).—The test results of metal-coated specimens by Mr. Fuller are of particular interest because of their confirming value and having been made on small size specimens.

Mr. Lessells inquires as to the reason for the increased endurance limit reported for the case of axles metal sprayed in the wheel fit portion for which we have no explanation. Possibly the sprayed material has a low susceptibility to the rubbing corrosion and stress concentration presented by press-fitted assemblies.

The influence of surface finish and differences in contacting conditions between the press-fitted wheel and axle are illustrated in the following table of endurance limit values established for $1\frac{3}{4}$ in. diameter axles of S.A.E. 1045 steel machined from $2\frac{1}{4}$ in. diameter hot-rolled bar stock without heat treatment:

	Allowable Maximum Axle Bend'ng Stress in Wheel Seat to Prevent			
	Initiation of Fatigue Cracks		Breaking off of Axle	
	psi.	per cent	psi.	per cent
Wheel pressed on axle—smooth turned surfaces	9 000	100	11 000	100
super finish on axle and wheel bore	10 500[a]	117	12 500	113
Wheel shrunk on axle—smooth turned surfaces	7 500[a]	83	9 500	86

[a] Tentative values.

The question of thickness of flame hardened case in relation to the fatigue resistance of the wheel seat of the 2-in. axles tested is one which was not investigated. It is our opinion, however, that the case depth used could be decreased with little or no change in the fatigue values obtained.

Fatigue tests are being made on induction-hardened axles where the design, material, and test conditions are identical to those followed with the flame-hardened axles. Preliminary results indicate that the fatigue values will be comparable with those obtained with the flame-hardened members.

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[4] Manager, Mechanics Division, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[5] In Charge of Railway Engineering and Research, The Timken Roller Bearing Co., Canton, Ohio.

FATIGUE OF POROUS METALS

By Claus G. Goetzel[1] and Richard P. Seelig[2]

Synopsis

Porosity, whether an unintentional feature or purposely produced, is expected to impair the physical properties of a given material. Some studies of the property changes of cast iron with graphite inclusions (which are comparable to pores) have been carried out by different authors.

The art of powder metallurgy offers a unique method by which to produce porous metals and alloys. Bars were pressed and sintered so as to contain controlled amounts of porosity. These bars were machined into fatigue specimens for the Moore and Wishart testing machine. *S-N* curves were recorded both for copper and iron and compared with those of cast copper and ingot iron, respectively. The fatigue results were supplemented by tension tests and a ratio of fatigue strength to tensile strength determined for different amounts of porosity. The results indicate that the fatigue limit of a given metal drops with the total amount of porosity. As long as this porosity is not excessive this drop is, however, less than expected.

On the other hand, the effect of mere reduction in cross-sectional area due to the pores is not entirely responsible for this. The pores exert certain external and internal notch effects.

Porosity in engineering materials in the majority of cases is a highly undesirable feature. This is generally accepted as being true although there is very little known about the actual effect of porosity on the physical properties of a given material. On the other hand, there are instances where porosity is purposely produced in metals. Here, too, the problem of physical properties as compared with dense material is of great importance.

Unintentional porosity occurs frequently in cast materials both ferrous and non-ferrous. Graphite laminations in cast iron may be considered as porosity, as they form an interruption in the metallic structure.

The importance of the problem in steel casting may be emphasized by the following example: In a certain steel foundry, according to Woodward (**21**),[3] where porosity had been perhaps more persistent than is usually experienced, a study of rejection records was made covering a period of 6 yr., 1927 to 1932, inclusive. A total of 21,057 castings were rejected on account of porosity.

Another material where porosity is often objectionable is bronze in the cast condition. Also chill-cast gun metal often forms pores and blowholes. Moreover, metal coatings sometimes show a porous structure. Thus, we see that there is a large number of instances where porosity plays an important part. It is surprising to find, therefore, that no systematic studies on the influence of pores on the physical properties, especially on the fatigue properties, have been made. Some interesting papers on related subjects will be reviewed briefly.

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[1] Assistant Director of Research, American Electro Metal Corp., Yonkers, N. Y.

[2] Plant Manager, Powder Metallurgy, Inc., Long Island City, N. Y.

REVIEW OF PREVIOUS WORK

Test results for special machined specimens of cast iron showed the interior metal to be slightly stronger in tension and of about the same strength in flexure as the outer layers (Schlick and Moore (**17**)).[3]

The physical properties of gray iron castings are affected to a greater extent by differences in section than those of other cast metals (Campbell (**2**)). In heavy sections which cool slowly in the mold the strength values are lower than in light sections which cool more rapidly. This is caused by differences in the proportion and condition of the graphite in the metal. Slow cooling promotes the separation of graphite and results in a decrease in the tensile strength of the metal. Similar results are reported from flexure tests with cast iron (MacKenzie and Donoho (**8**)). In the investigation with cast iron, Draffin and Collins (**3**) showed that abrupt decreases in section appear to weaken the specimen more than surface defects. Relatively little difference was found between the strength of solid and hollow specimens although the solid ones were slightly stronger. Surface finish is not particularly important in tension. Small superficial differences do not produce stress concentrations which would greatly affect the strength.

According to Thum and Ude (**20**) cast iron is considered nothing else but notched steel. Thus many deviations from the properties of steel can be explained. The properties can be compared to those of steels when taking the notch effect of the graphite veins into account. The relatively high notch resistance of cast iron under fatigue stress is ascribed to the notch effect of microscopically fine graphite laminations (internal notch effect). They decrease the fatigue limit to such an extent that artificial notches and rough surface no longer show any influence.

Tension, compression, and impact tests by Thum and Ude (**20**) have shown that cast iron may be considered as steel with holes or cavities which represent the graphite inclusions. Steel specimens with holes or slits were found to have physical properties very similar to those of cast iron. It is very interesting to compare the relationship between tensile results and fatigue results for different metals. Figured from results of Pomp and Hempel (**15**) the ratio of fatigue strength under repeated compression to fatigue strength under repeated tension was 2.7, 4.2, and 3.4, respectively. The ratio of plain compressive strength to plain tensile strength was found to be 3.5, 4.2, and 3.8, respectively. This seems to be new proof that the internal notch effect of graphite inclusions is less pronounced under compression. Gough (**5**) finds that cast materials may differ considerably one from the other in their tensile properties, while they may have relatively constant ratios of fatigue to tensile strengths. The fatigue strengths were found to be higher than expected in relation to the tensile strengths, especially considering possible casting defects. The results of experiments by Mailänder (**9, 10**) are shown below:

Material	Tensile Strength, kg. per sq. mm.	Fatigue Strength, kg. per sq. mm.		Decrease by Notch Effect, per cent	Ratio, Fatigue Strength to Tensile Strength	
		Plain	Notched		Plain	Notched
Cast iron	21	9	9	0	0.43	0.43
Cast iron	27	11.5	11.5	0	0.43	0.43
Centrifugal casting	45	18	16	11	0.40	0.36
Steel	44	23	15.5	32	0.52	0.35

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 755.

The more graphite cast iron contains and the coarser it is the lower is the tensile strength and the lower (and more dependent on stress) is the modulus of elasticity. For machined specimens the ratio of fatigue strength (in bending) to tensile strength was found to be 0.45 (Mailänder (**9**, **10**)). It is not much lower than that for steel. In other words, the notch effect of graphite decreases not only the fatigue strength but also the tensile strength as compared with ductile steel.

Another indication for the internal notch effect of graphite is seen in the fact that the ratio of torsion fatigue limit to bending fatigue limit is higher for cast iron than for steel. A similar high ratio of torsion fatigue limit to bending fatigue limit (0.91) for magnesium and aluminum casting alloys is blamed by Matthaes (**11**) on pores. This seems to be a reasonable assumption, as aluminum castings are less notch sensitive than aluminum forgings. It is a well-known fact that all irregularities of the specimen surface have a more or less detrimental influence on the fatigue properties of metals. When determining the effect of such irregularities it is necessary of course to standardize on an ideally smooth surface (for instance, polished and buffed). On the other hand, this comparison may not be in all cases of practical value, because ideally smooth surfaces are not found in structural parts. Therefore, the influence of small surface defects on aluminum wires was compared with the ordinary drawn surface of aluminum wires in previous tests by one of the authors (**18**). In this comparison the influence of the notches was not very great. In this connection a most interesting research by Kenyon (**7**) should be mentioned. He found that the addition of 4 per cent lead has little effect on the endurance properties of a tin-base bearing alloy. In fact, the lead seems to improve the material slightly. This is interesting because current belief is that lead has an embrittling effect on the alloy and that lead produces minute shrinkage cavities in such alloys. Specimens with visible defects, on the other hand, were very much inferior to sound metal.

The only fatigue tests carried out with sintered metals so far are reported by one of the authors (**4**).

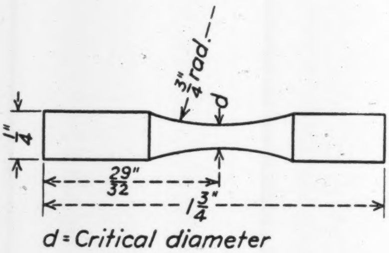

FIG. 1.—Fatigue Test Specimen for Moore-Wishart High-Speed Machine.

EXPERIMENTAL WORK

Powder metallurgy is unique in that it is possible to produce materials of controlled porosity. Therefore, this method was used to produce specimens for this investigation. Bars measuring $\frac{3}{8}$ by $\frac{3}{8}$ by 3 in. were compressed from copper and iron powder, respectively. The variations in percentage porosity were achieved by variation in compacting pressure and the size of the pores was varied by using fine or coarse grades of powders. After compression the bars were then sintered in hydrogen gas and a few of them were compressed and sintered a second time after the sintering to produce lowest porosity. The materials used, the pressing and sintering treatment, and the resulting porosity are shown below:

Specimen Set Number	Basic Material	Processing Method for Specimens[a]		Porosity, per cent
		Pressure, tons per sq. in.	Sintering Cycle	
A 1....	Electrolytic copper powder of over 200 mesh	15 (1×)	750C./16 hr./1×	23.5
A 2....		50 (1×)	750C./16 hr./1×	6.5
A 3....		50 (2×)	750C./16 hr./2×	3.5
C 1....	Electrolytic copper powder of less than 325 mesh	15 (1×)	750C./16 hr./1×	22.5
C 2....		50 (1×)	750C./16 hr./1×	7.0
C 3....		50 (2×)	750C./16 hr./2×	4.5
D 1....	Electrolytic iron powder between 100 and 200 mesh	15 (1×)	1000C./8 hr./1×	32.0
D 2....		50 (1×)	1000C./8 hr./1×	20.8
D 3....		50 (2×)	1000C./8 hr./2×	10.0
F 1....	Electrolytic iron powder of less than 325 mesh	15 (1×)	1000C./8 hr./1×	32.5
F 2....		50 (1×)	1000C./8 hr./1×	18.3
F 3....		50 (2×)	1000C./8 hr./2×	6.0

[a] 1× and 2× indicate whether pressed and sintered once or twice. Furnace atmosphere was dry hydrogen.

The apparent density was determined on the square bars by measuring the volume and determining the weight. The porosity was computed by comparison with the theoretical density for either metal. The fatigue specimens were machined out of the square bars according to the shape and size required for the Moore and Wishart fatigue testing machine (see Fig. 1). Standard tension test specimens were also compressed and sintered by the same procedure so that the tensile strength could be determined for each kind of porosity on both metals. The fatigue tests were run on the Moore and Wishart machine and *S-N* curves were plotted. A series of photomicrographs was taken of all classes of porosity both in copper and iron. See Fig. 2 and 3.

The purpose of micrographs is twofold: they show the amount and nature of the pores; they also tell something about the pore distribution. It was suspected that due to turning and handling, the pores near the circumference of the bars might be closed or dislocated. For this reason two micrographs were taken of each bar, one in the center and one at the surface. Our assumption was found to be true to some extent. The highly porous specimens show some dislocation of material near the surface. It must be concluded that this is so slight that it does not interfere with the results. The denser specimens show no dislocation of this kind; the pores near the surface are not closed up.

The resultant fatigue strength values together with tensile strength values and the ratio of both properties are shown below:

Specimen Set Number	Fatigue Strength, psi.	Average Tensile Strength, psi.	Ratio, Fatigue Strength to Tensile Strength, per cent
A 1.........	3 700	10 770	34.2
A 2.........	7 500	18 870	39.8
A 3.........	10 500	25 200	41.8
C 1.........	4 700	13 000	36.2
C 2.........	9 500	23 180	41.0
C 3.........	13 000	30 000	43.3
D 1.........	4 000	12 500	32.0
D 2.........	9 000	26 500	33.3
D 3.........	14 500	34 900	42.7
F 1.........	4 500	13 350	33.7
F 2.........	11 500	31 900	34.5
F 3.........	23 000	37 700	61.0

DISCUSSION OF TEST RESULTS

Fatigue curves for two sets of solid and six sets of porous copper specimens are shown in Fig. 4. Hard-drawn oxygen-free high-conductivity copper shows of course the highest curve. Soft annealed oxygen-free high-conductivity copper has a lower fatigue limit than three of the porous specimens.

It is interesting to note that sample C 3 has a fatigue limit which is 2500 psi. higher than A 3 although the porosity for C 3 is slightly greater. Referring to Fig. 2, this is easily explained (photo-

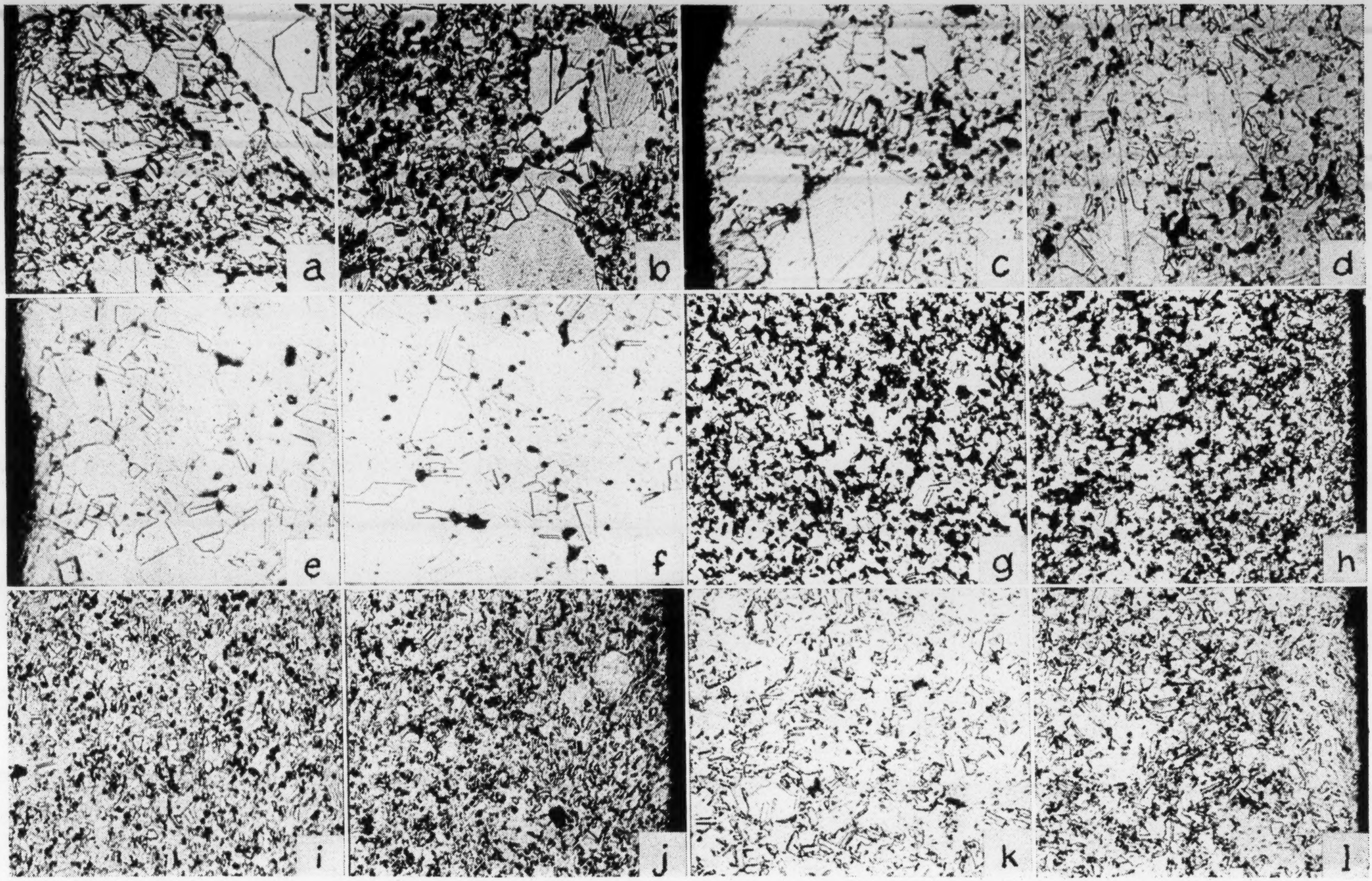

FIG. 2.—Microstructure of Sintered Copper Specimens (×200).

(*a*) Specimen of Set A 1 (edge), 23.5 per cent porosity.
(*b*) Specimen of Set A 1 (center), 23.5 per cent porosity.
(*c*) Specimen of Set A 2 (edge), 6.5 per cent porosity.
(*d*) Specimen of Set A 2 (center), 6.5 per cent porosity.
(*e*) Specimen of Set A 3 (edge), 3.5 per cent porosity.
(*f*) Specimen of Set A 3 (center), 3.5 per cent porosity.
(*g*) Specimen of Set C 1 (center), 22.5 per cent porosity.
(*h*) Specimen of Set C 1 (edge), 22.5 per cent porosity.
(*i*) Specimen of Set C 2 (center), 7.0 per cent porosity.
(*j*) Specimen of Set C 2 (edge), 7.0 per cent porosity.
(*k*) Specimen of Set C 3 (center), 4.5 per cent porosity.
(*l*) Specimen of Set C 3 (edge), 4.5 per cent porosity.

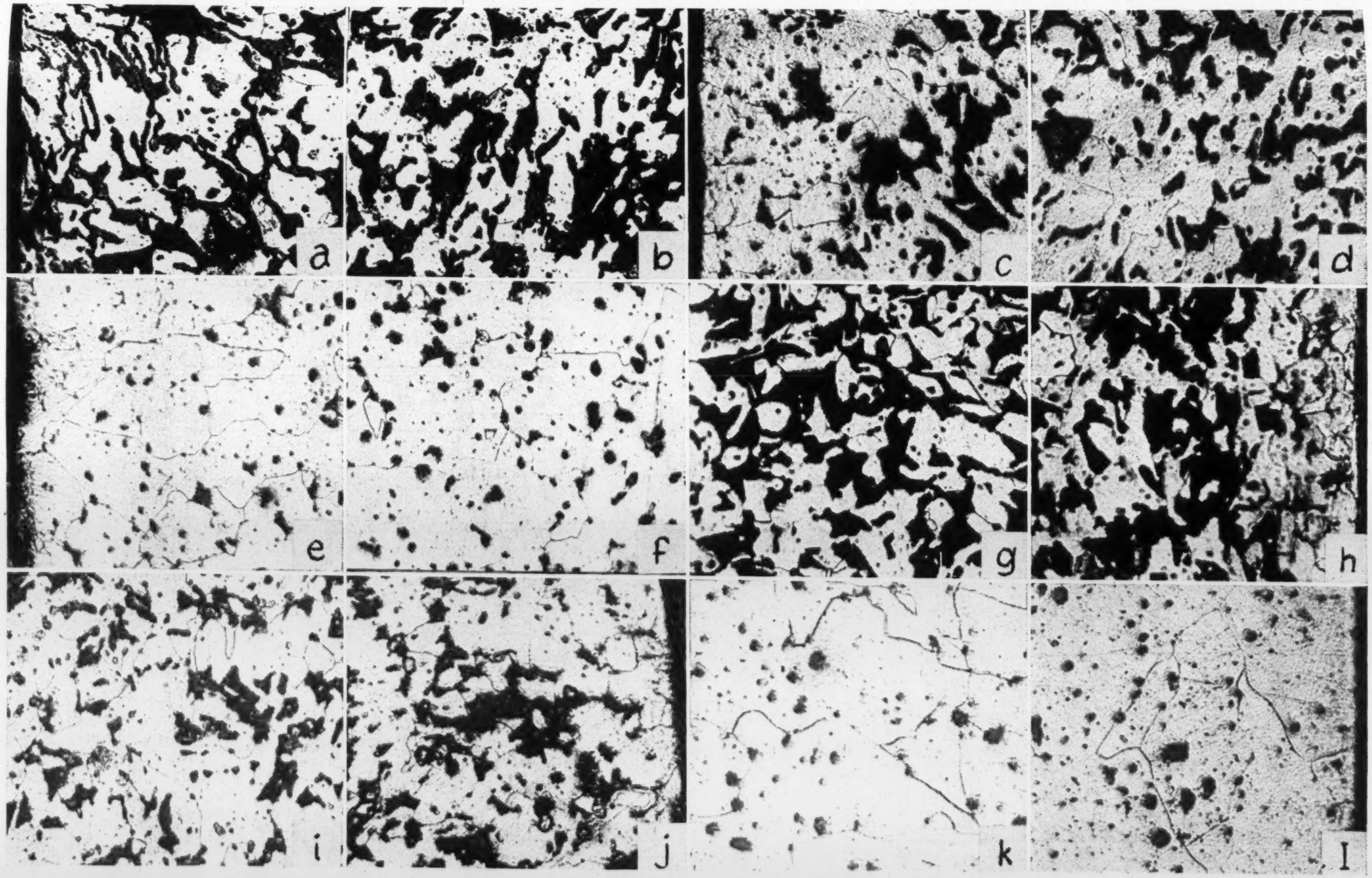

FIG. 3.—Microstructure of Sintered Iron Specimens (×200).

(*a*) Specimen of Set D 1 (edge), 32.0 per cent porosity.
(*b*) Specimen of Set D 1 (center), 32.0 per cent porosity.
(*c*) Specimen of Set D 2 (edge), 20.8 per cent porosity.
(*d*) Specimen of Set D 2 (center), 20.8 per cent porosity.
(*e*) Specimen of Set D 3 (edge), 10.0 per cent porosity.
(*f*) Specimen of Set D 3 (center), 10.0 per cent porosity.
(*g*) Specimen of Set F 1 (center), 32.5 per cent porosity.
(*h*) Specimen of Set F 1 (edge), 32.5 per cent porosity.
(*i*) Specimen of Set F 2 (center), 18.3 per cent porosity.
(*j*) Specimen of Set F 2 (edge), 18.3 per cent porosity.
(*k*) Specimen of Set F 3 (center), 6.0 per cent porosity.
(*l*) Specimen of Set F 3 (edge), 6.0 per cent porosity.

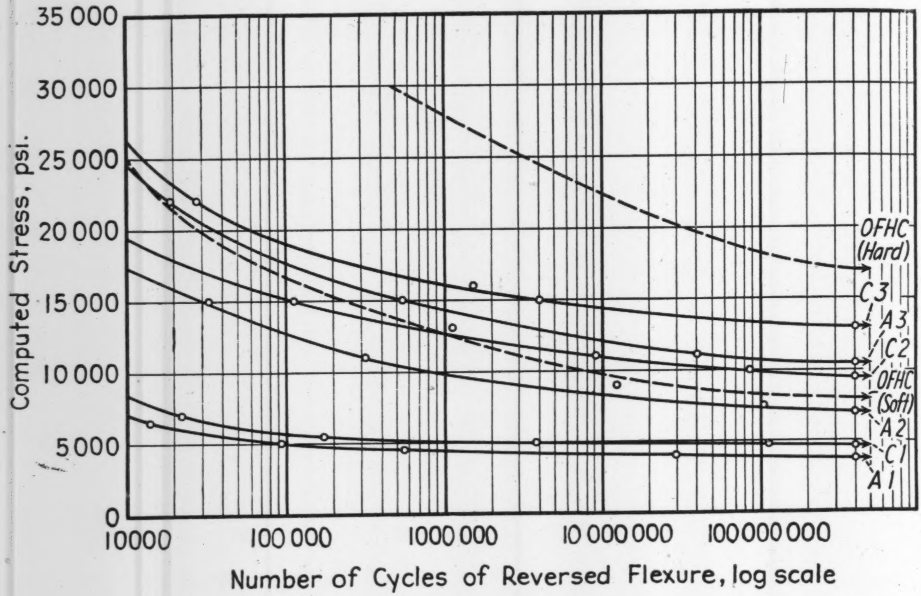

FIG. 4.—Fatigue Properties of Sintered Copper of Different Porosity.
OFHC = oxygen-free high-conductivity copper.

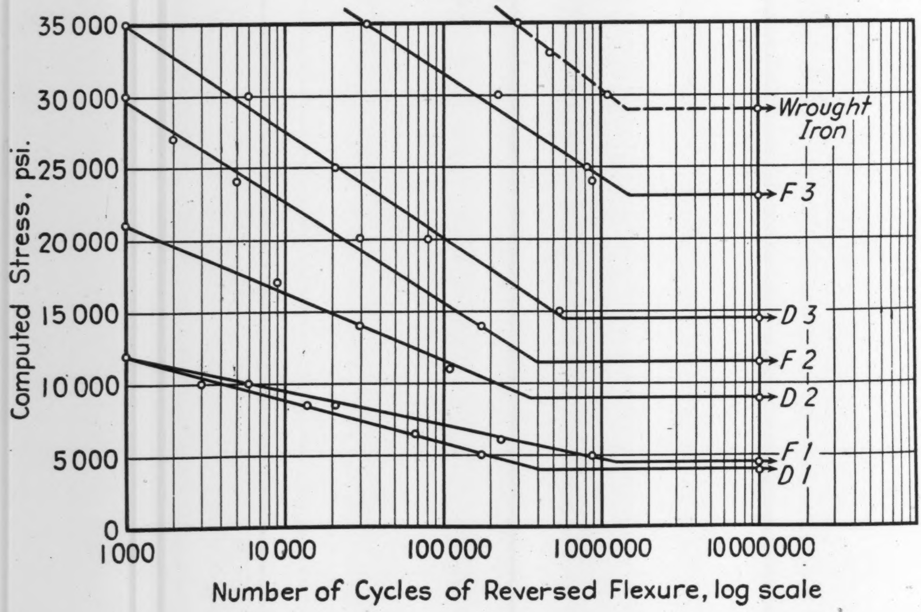

FIG. 5.—Fatigue Properties of Sintered Iron of Different Porosity.

micrographs *e*, *f* and *k*, *l*, respectively). The porosity of C 3 is more finely subdivided than that of A 3.

In this one example the influence of the pore size seems to be demonstrated. On the other hand, this influence cannot be too strong, because in no case does coarse porosity exert a more predominant influence than that of total pore percentage. It may also be noted that the difference between two types of porosity (series A and C) disappears with increasing total porosity. Not too great importance should be attributed to this because as may be seen from the photomicrographs the size of the pores does not appear to be a great deal different in the highly porous specimens.

In Fig. 5, similar curves are shown for one series of ingot iron and six sets of porous iron specimens. The solid iron specimen shows the highest fatigue curve. A similar condition prevails as in the case of copper. The fatigue strength suffers with increasing porosity. It may be pointed out that the difference between the specimens prepared from fine and coarse powder is greater here than in the case of copper. As may be seen by a comparison of the photomicrographs in Fig. 3 and the corresponding porosity values this may be due to the difference in porosity percentage rather than in pore size. With increasing total porosity the difference in fatigue properties between the two types of specimens disappears.

The *S-N* curves run less steep with increasing porosity both for copper and iron. The curves for the powder pressed copper specimens run flatter than those for oxygen-free, high-conductivity copper, both in the hard-drawn and soft condition. For iron the slope of the curves of the sintered specimens is pretty well in line with that of ingot iron. The curves for porous copper specimens are smoothly curved just like the ones for solid copper. The porous iron specimens show a turning point similar to that which solid iron specimens usually have.

A comparison of the authors' results with cast iron is not shown in the curves. Using the figures given by Mailänder (9, 10) it is found that the fatigue strength of low-grade cast iron is lower (12,800 psi.) than that of the powder pressed and sintered iron specimens with 6 and 10 per cent porosity, respectively. As a matter of fact, this type of cast iron corresponds roughly to a sintered specimen with 15 per cent porosity. A higher grade cast iron (with a fatigue strength of 16,200 psi.) corresponds to about 12 per cent porosity in a specimen made from fine iron powder and to about 8.5 per cent porosity in a specimen made from coarse iron powder.

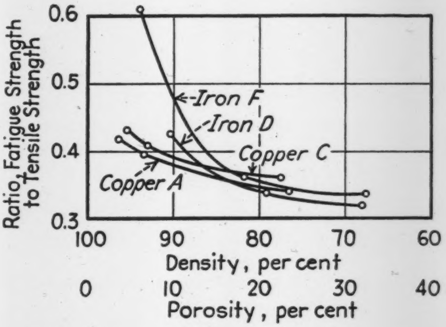

FIG. 6.—Fatigue-Tension Ratio as a Function of Porosity for Copper and Iron.

The fatigue strength of cast steel exceeds that of any of our sintered specimens. However, a notched steel specimen, according to Mailänder (9, 10), has a fatigue strength that is a little lower than that of our series F 3, made from fine iron powder with a total porosity of 6 per cent.

These comparative data with cast iron and steel are supplementary to the iron curves. They are based on averages and extrapolated data. To be sure, it should be noted that the structure of cast iron is very different from sintered (pure) iron. The values are approximate only but should be of considerable practical interest.

It is interesting to study the ratios of fatigue strength to tensile strength, see Fig. 6. This ratio is very high for high-density iron and drops rapidly as the porosity increases. For copper it does not drop so drastically and for high porosity it is higher than for similar iron specimens. One of the most important findings of this investigation appears to be the fact that these values, plotted against porosity, form smooth curves in all cases.

It may also be noted that the curves for fine powder (small pores) lie consistently higher than for coarse material both in the case of iron and copper. This is a very interesting observation as it is the second instance we find where the particle size exerts a slight but definite influence.

A comparison with the figures of Mailänder quoted earlier shows that the ratio of fatigue strength to tensile strength for cast iron corresponds approximately to the ratio of the sintered specimens with about 9 per cent porosity in the case of coarse iron powder, or 11.5 per cent porosity in the case of fine iron powder. The figure for plain steel would correspond to a porosity of approximately 5 per cent in the sintered specimens when extrapolating the two iron curves (Fig. 5) and taking an average value of these two. The ratio for the notched steel specimen as reported by Mailänder reproduced above, would correspond to 19 per cent of pores in the sintered specimens.

Evaluation of Results and Theory:

Irregularities of specimen surfaces and notches may be termed technological danger points. With static load, that is, one-directional single-stroke load, the notch effect is not very dangerous. Tougher materials are able to eliminate stress-raising effects by plastic deformation. In fact, there are cases where notches increased the static tensile strength, for instance, in the case of cast iron (Sachs (**16**)).

As is well known, this is in no case true when changing or oscillating load is applied. For instance, even the slightest uneveness of surface impairs the fatigue properties; it is incorrect to consider the fatigue strength a strictly defined material property. It is a value that is determined by the material, the specimen shape, the surface condition and, as we have seen, the porosity.

It is generally accepted that the influence of notches is based on the stress concentration in the bottom of the notch. The same is probably true for pores. Attempts were made by means of formulas to explain the stress-raising effect. If we call the increase in stress s, depth of indentation d, the radius of curvature in the root of the notch r, the following relations were found:

Griffith $$s = \left(1 + \sqrt{\frac{d}{r}}\right) \times 100 \text{ per cent}$$

Inglis $$s = \left(1 + 2\sqrt{\frac{d}{r}}\right) \times 100 \text{ per cent}$$

Armbruster $$s = \frac{2y + 4\sqrt{y} + 2}{2 + \sqrt{y}}$$

The latter formula applies to half elliptical notches in which $y = \frac{d}{r}$ (see Armbruster (**1**)).

All these formulas are admittedly only very approximate. They apply to tensile stress on a specimen the width of which is very large as compared to the depth of the notch.

A practical formula which has to be

adapted to each particular condition was developed by Thomas (**19**):

$$\sigma = \sigma_m\left(1 + K\sqrt{\frac{d}{r}}\right).$$

Herein σ is the maximum strength in the notch bottom and σ_m the mean tensile strength. For sharp notches $K = 2$, for fine grooves $K = 0.16$, and for coarse notches $K = 0.45$.

Another approach to the problem of the influence of notches was the experimental determination by means of optical polarization with glass rods, by means of very accurate extensometer measurements, and by means of rubber models. In a comparison it is found that the results of the different investigators are not in agreement.

Since it must be stated that these formulas and experimental data may not be satisfactory for practical determination of notch effect, it is at present not obvious how they can be translated into terms of internal porosity. The effect of pores on the stress distribution may be visualized in a twofold manner. The stress in the porous cross-section is increased in accordance with the ratio of the moments of resistance for the original and the reduced cross-section. Moreover the stress-raising effect similar to the notch effect has to be considered.

From our curves it may be concluded that the reduction in cross-sectional area between pores alone cannot be responsible for the reduction in fatigue limit. On the other hand, the stress-raising effect which was expected to reduce the fatigue limit to a greater extent seems relatively low. It would seem probable from experiments on multiple stress raisers closely spaced together that a general porosity might reduce the stress concentration at each individual pore.

The results described indicate that the condition of a porous fatigue specimen is characterized both by reduction in cross-sectional area and some degree of stress-raising influence. No qualitative conclusion as to the degree of influence of these features can be drawn at the present time.

In conclusion, it should be mentioned that the fatigue strength of the sintered specimens is relatively high as long as the porosity is not excessive. The influence of corrosion has not been covered by this investigation. It is quite possible that corrosion-fatigue phenomena are more pronounced in porous specimens than they are in solid ones.

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(3) J. O. Draffin and W. L. Collins, "The Tensile Strength of Cast Iron," *Proceedings,* Am. Soc. Testing Mats., Vol. 37, Part II, p. 88 (1937).

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(13) H. F. Moore, Private communication.

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(16) G. Sachs, "Über Kerbwirkungen beim Stauchversuch," *Stahl und Eisen*, Vol. 43, II, p. 1587 (1932).

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DISCUSSION

Mr. J. B. Kommers[1] (*presented in written form*).—This paper is of much interest because it gives information on fatigue strength of new materials.

In the accompanying Fig. 1 I have plotted endurance limit and also tensile strength against porosity. It is of interest to note that the endurance limit does not vary linearly with porosity. As the porosity increases the endurance limit decreases at a reduced rate.

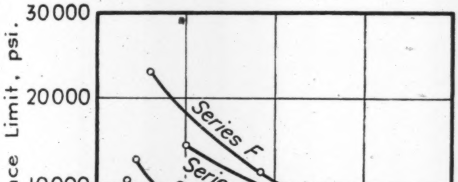

Fig. 1.—Relation of Endurance Limit and Tensile Strength to Porosity.

For the tensile strength the same type of curve is obtained for the copper specimens as was found for all the endurance curves. However, this is not the case for the tensile strength of the iron specimens. For the latter it will be noted that the tensile strength decreases at an increasing rate with increase in porosity. This indicates how difficult it is to predict the endurance behavior of new materials. It reinforces the argument for experimental work.

If Figs. 4 and 5 represent all the fatigue results obtained on these materials, it should be noted that all the curves show rather remarkable uniformity of results. This is evidenced by the lack of scatter of the fatigue results.

Mr. J. T. MacKenzie[2]—I was interested in this paper from the standpoint of cast iron, which acts when under stress very much like a porous material and therefore I went over the cast irons of the 1933 impact reports and plotted the fatigue strength against the volume percentage of graphite and then put Goetzel and Seelig's irons on the same graph (accompanying Fig. 2). It is interesting to observe how with the low values the two materials check, but at the 10 per cent point the truly porous material is very much stronger than the graphite bearing material. The graphite does not act as a porous material in compression, and the chances are that the stresses are higher than are calculated due to this; also, the matrix of the high graphite irons is quite weak.

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[1] Professor of Mechanics, Engineering College, University of Wisconsin, Madison, Wis.

[2] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

MR. R. E. PETERSON[3] (*by letter*).—It is of interest to examine the data in the light of theoretical stress concentration values for enclosed cavities and for surface discontinuities. For a spherical cavity in a tension member Goodier[4] has found a theoretical stress concentration factor of approximately 2. This is lower than for a small circular hole in a flat tension bar which approaches 3 as a limit. It therefore appears that failure would start at the surface where one of the voids has been cut rather than at one of the voids just below the surface.

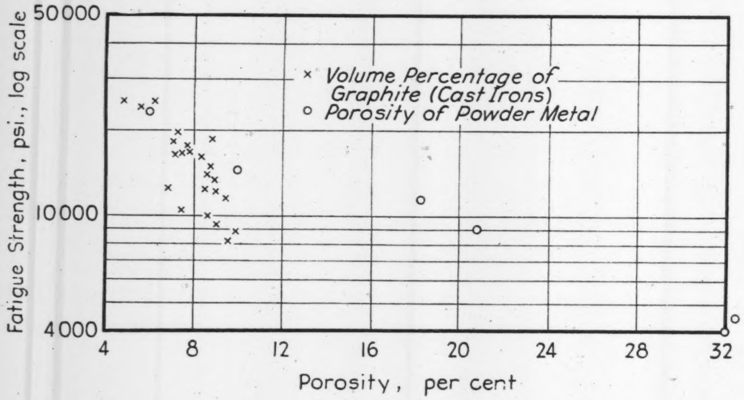

FIG. 2.—Relation of Fatigue Strength to Volume Percentage of Graphite.

If we now consider the authors' data, iron without cavities has an endurance limit of approximately 29,000 psi. and with 32 per cent porosity this drops to 4000 psi. Due to porosity the reduction of net area would reduce the section modulus to where one might expect about 20,000 psi. endurance limit if no stress concentration effects were present. We now see that a stress concentration factor of 5 is needed to reduce this figure to 4000 psi. It therefore appears that spherical cavities cannot produce such a result. However, examination of the micrographs of the specimens having large porosity shows some of the pores to be quite elongated.

Let us now refer to Neuber's results[5] for a tension member having a cavity generated by an ellipse with its minor axis coinciding with the axis of member. The stress concentration factor, K, depends on t/r where t = radius of the cavity about the axis and r = maximum radius at the end of the cavity in an axial plane. For $K = 5$, a value of $t/r = 10$ is needed. If we consider a corresponding elliptical hole in a flat tension member, the concentration effect is apparently greater so that a t/r of but 4 is needed. As before, surface failure is to be expected. It is certain however that a still larger t/r, that is, a sharper discontinuity, will be required for the following reasons:

(*a*) The above value is, in each case, for an isolated cavity in a large body.

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[3] Manager, Mechanics Division, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[4] J. N. Goodier, "Concentration of Stress Around Spherical and Cylindrical Inclusion and Flaws," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 55, APM-55-7, p. 39 (1933).

[5] H. Neuber "Kerbspannungslehre," p. 99, Julius Springer, Berlin (1937).

For a porous material made up of cavities rather closely spaced, a higher t/r would be needed to give the same K.

(*b*) Where the size of discontinuity is of the same order as the grain size, the loss of sensitivity[6] is large. For this reason a higher t/r would be needed to arrive at a final fatigue limit of one-fifth the nominal value. Taking the foregoing considerations into account one would need an extremely narrow form of discontinuity, say of the order of t/r of 10, having the appearance of a crack, to account for the results.

One observation which follows from the foregoing analysis is that if the pores could by some modification of the process be made to be of more nearly spherical form, as seems to be the case for the materials of low porosity, greatly increased fatigue strength could be expected.

It is hardly necessary to add that the paper by Messrs. Goetzel and Seelig constitutes an important addition to the literature on fatigue of metals.

MR. J. M. LESSELLS.[7]—The paper is interesting in that it represents a step towards telling us something about the mechanical strength obtained by powder metallurgy. So far powder metallurgy has been confined to the domain of the metallurgist and physicist but this is the first serious attempt to give the engineer some data on the strength to be expected by the use of this process.

It is likely that the pressure applied in the process will have a great influence on the results obtained, and possibly the mechanics of how we get a solid body by the process is not as yet clearly understood. It must be true that very high temperatures are generated as a result of the pressures employed, and possibly the pressure variable should be considered.

MR. L. W. BALL.[8]—In any consideration of porous metal, I think the relation of the structure to radiographic appearance should be brought out in the discussion. In radiographic testing we have a good many types of mottled appearance on our film, due to very small gas cavities between the grains of metal, and this is particularly true in the aluminum alloys and also in magnesium alloys. There have been at least two sets of research in which the fatigue strength of porous metal has been related to radiographic appearance. We have not measured the porosity in terms of per cent but rather in degrees of the mottling on the film, and the relation between the fatigue strength and the mottling is very direct. Mr. Triplett, I believe, has prepared curves relating the two. It would have been of considerable interest to the radiographers if the specimens in the present investigation had been radiographed.

MR. FRANCIS M. HOWELL.[9]—In the Aluminum Research Laboratories we have made fatigue tests on sand-cast aluminum alloys having different degrees of porosity. We have found that while, in general, there is a relation between the amount of porosity and the endurance limit, it is dependent not only upon the amount of porosity, but also upon the location of the porosity, the shape of the voids and many other things, so that we do not believe that by determining only the percentage of porosity, the effects of porosity upon the endurance limit can be determined.

MR. CLAUS G. GOETZEL.[10]—Mr. Lessells mentioned that the pressure might promote extensive heating which might have something to do with the results of the test. Some heat might be created during compression, but only a cohesive force comes into action, which has to be replaced by atomic forces, that is, by diffusion, during heat treatment. The pressure's primary effect, however, was found to be the compacting of the particles to bring them into an agglomerated condition. What is most important is not the pressure but the heat treatment, because during this sintering process we go through a metamorphosis of our material. At the beginning we have hundreds of thousands of small particles of low tensile strength, and only after heat treatment which promotes grain growth do we obtain a metallic body which may be compared with cast metal, though the difference in grain size is of important influence.

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[7] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[8] Division of Physics and Electrical Engineering, National Research Council of Canada, Ottawa, Ont., Canada.

[9] Engineer, Aluminum Research Laboratories, Aluminum Co. of America, New Kensington, Pa.

[10] Assistant Director of Research, American Electro Metal Corp., Yonkers, N. Y.

We found a certain difference in the curves for the fine and coarse materials. For instance, in the case of iron, the F 3 curve is well above the D 3 curve. That might be partly due to the fact that the porosity was slightly lower in the one set of specimens than in the other. If one compares the pictures, one will see that the micrographs of specimen F 3 (Fig. 3(*k*) and (*l*)) show a larger grain size than those of specimen D 3 (Fig. 3(*e*) and (*f*)). There are more grains coming in contact with each other in the case of D 3 than in the case of F 3. This appears to be one of the most important factors governing the tensile strength as well as the fatigue strength, because evidently in our, may I call it, "synthetic" material, made by powder metallurgy, the weak planes are in the grain boundary areas. The grain boundaries, created in the heat-treating process are artificial and weakened by impurities and oxides. Therefore it seems that the fewer boundaries we have in the material, the better our metal will be with regard to tensile strength and fatigue strength.

An explanation for the peculiar fact that the grain size of material made from the fine powder was larger than that made from the coarse powder cannot be assumed so easily; the fact that we used very pure iron powder might have caused a more spontaneous grain growth in the fine powder material. Besides, sintering was carried out at a temperature very critical for grain growth phenomena of sintered iron, and slight temperature variations (in the range of 10 C.) might have had some effect.

Mr. MacKenzie mentioned that cast iron behaves very well under compression and not so well under tension. We have observed exactly the same fact with powder metals. They usually stand up very well under compression, but when it comes to tension they are rather weak, so that the comparison holds.

I agree with Mr. Howell, that the shape of the voids makes quite a difference; that is the reason why we showed detailed photomicrographs so that one can see the difference in the shape. The shapes of these pores can be controlled by the size of the particles and also by the pressure and method of heating.

I agree with Mr. Ball that radiographic investigation would probably throw even more light on the question of the nature of the pores, but unfortunately such facilities were not available, so that the microscope had to be used instead.

MR. RICHARD P. SEELIG.[11]—Mr. Howell pointed out that the location of the pores is an important factor.

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[11] Plant Manager, Powder Metallurgy, Inc., Long Island City, N. Y.

From Mr. Peterson's discussion it is evident that the shape of the pores also has to be considered. However, in order to start a systematic research on the influence of porosity on mechanical properties, we decided to use porosity that was distributed as evenly as possible throughout the cross-section because by the use of our method this is a reproducible structure. It seems likely that conclusions may be drawn from this type of porosity and applied in other cases where no such homogeneous distribution exists.

Appreciation is expressed to Messrs. Kommers and Peterson whose discussions are a most valuable addition to the paper.

A PULSATING TENSION-FATIGUE MACHINE FOR SMALL DIAMETER WIRE

By John N. Kenyon[1]

Synopsis

A new type of pulsating tension-fatigue machine is developed by employing three reciprocating forces, 120-deg. out of phase, which exerts a constant torque on the motor and thus eliminates unbalanced inertial effects. A gripping method is developed whereby over 80 per cent of the test specimens fail clear of the end connectors. The machine is especially adapted for testing used wire in order to determine the extent of deterioration in endurance properties.

While corrosion-fatigue tests, performed on selected wires, give valuable information, the determination of the endurance properties of samples taken from service by a stress-reversal method are difficult because of the curved and "kinked" conditions of these wires. For such wires, not suitable for tests by stress reversal, a pulsating tension-fatigue test is more desirable. While the design of the necessary equipment offers some mechanical difficulty, the pulsating tension test has certain advantages in that the entire cross-section of the test specimen is nominally subjected to the desired maximum stress. This most effectively integrates the combined effects of surface imperfection and crystalline inhomogeneity.

Development of a Pulsating Tension-Fatigue Machine

The pulsating tension test described in this paper is essentially a method whereby a wire is subjected to some mean force, W, and this force rapidly varied from $W - w$ to $W + w$; the range of force is governed by the magnitude of $2w$. The purpose of the mean load W is to retain a tension load on the wire so that it will not buckle.[2]

Assume a crank and weight assembly wherein a body, E, suspended by a long wire, is accelerated upward and downward. This body moves with practically simple harmonic motion and subjects the wire to a pulsational tension force. If the crank is turned too fast, this wire will buckle when the accelerations of the body E exceed that of gravity. See Fig. 1 showing an experimental model.

Assume now that there are three bodies, A, B, and C, and that they are suspended by wires, 120-deg. apart, from the edge of a horizontal disk. From these three bodies, by means of a spider arrangement, is suspended a fourth weight D. When the disk is oscillated, by means of a crank, any point in the perimeter travels in a small vertical circle. The three weights are thus accelerated vertically in simple harmonic motion; should the accelerations exceed that of gravity, the wires will not buckle since they are held taut by the constant load D.

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[1] Instructor in Civil Engineering, Columbia University, New York, N. Y.

[2] Nomenclature for Various Ranges in Stress in Fatigue, Appendix, Report of Research Committee on Fatigue of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I, p. 159 (1937).

It can be shown that the three reciprocating movements, represented by A, B, and C, are actually a dynamically balanced force system with the driving mechanism exerting a constant torque. According to the principle of simple harmonic motion, the acceleration of a body is proportional to the distance from the center of oscillation. Let the three points of support on the disk be represented as constantly accelerating toward the center line of rotation $O-O'$ of Fig. 2. The equation of the motion is as follows:

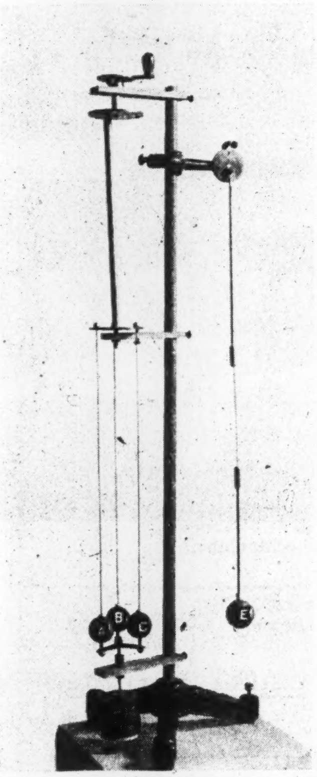

FIG. 1.—Pulsating Tension Experimental Model.

$$\text{Force} = \text{Mass} \times \text{Acceleration}$$

$$= \frac{W}{g} \frac{(2\pi)^2}{T^2} (x)$$

where W = weight of body,
T = period of oscillation, and
x = vertical distance from O to O'

At a given moment, the point of support for body A is at the lower limit of travel and with an upward acceleration proportional to $+x$. Simultaneously, bodies B and C, which are moving 120 deg. out of phase, have downward accelerations proportional to $-x/2$.

$$\text{Resultant force} = (m_A + m_B + m_C) \frac{(2\pi)^2}{T^2} (+x - x/2 - x/2) = 0$$

The force component can thus be shown to be zero for any position of the three bodies, hence the support of the disk exerts a constant upward force equal to the weight of $A + B + C + D$.

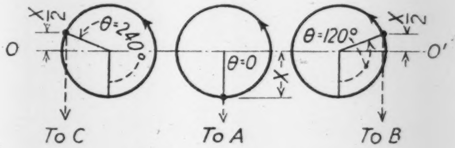

FIG. 2.

It has thus been shown that the three reciprocating forces reduce to a rotating force of constant torque and a balanced force system is secured. It is to be further noted, since body D has no motion and the three accelerations cancel, that each body may be treated independently as though it supported a nonreciprocating load $D/3$. Body A may be attached to the test specimen and the other two bodies B and C considered as counterweights.

CALIBRATION AND APPLICATION

The pulsating tension machine finally developed is identical in principle to the experimental model except that the reciprocating weights operate horizontally and the mean load D is supported by a right-angle lever, see Figs. 3 and 4. The horizontal type of machine was built because of simplicity of design and low cost, although it was realized that the traveling weights must be held in alignment by guides and a frictional effect thereby introduced.

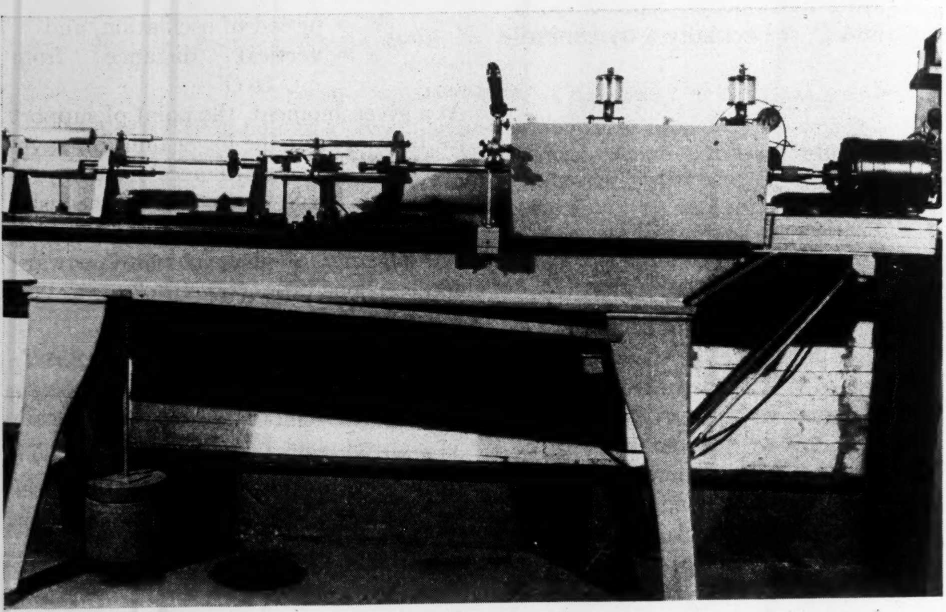

FIG. 3.—Pulsating Tension-Fatigue Machine.

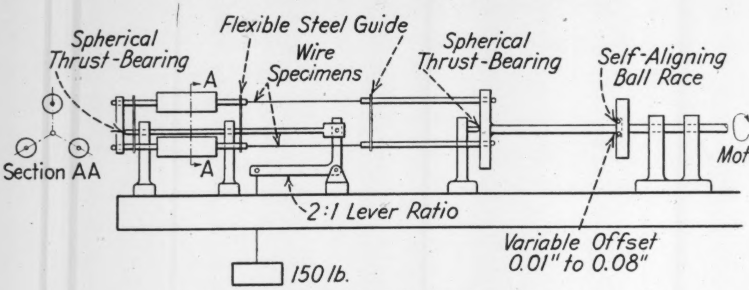
Spherical Thrust-Bearing
Flexible Steel Guide
Wire Specimens
Spherical Thrust-Bearing
Self-Aligning Ball Race
A
A
A
Section AA
Motor
2:1 Lever Ratio
Variable Offset 0.01" to 0.08"
150 lb.

FIG. 4.—Pulsating Tension-Fatigue Machine.

The machine is driven by a ½-hp., 3600-r.p.m. synchronous motor. The reciprocating weights are held in position by thin flexible steel guides which permit longitudinal movement and reduce friction to a minimum. Points of movable contact are fitted with ball bearings. The distance traveled by the reciprocating weight, to which the wire test specimen is attached, is determined by the Brinell micrometer microscope. This instrument is graduated to 0.1 mm. (0.004 in.) but by careful manipulation was estimated to one-fourth of a division (0.001 in.).

The following is an example of the inertial force exerted by a reciprocating weight:

$$F = \pm \frac{W}{32.2} \frac{(2\pi)^2}{T^2} \frac{(x)}{12}$$

$$= \pm \frac{4.5 \times 39.5 \times (0.052/2)}{32.2 \times (1/60)^2 \times 12}$$

$$= \pm 1660 \times 0.052/2$$

$$= \pm 43 \text{ lb.}$$

where Speed = 3600 r.p.m. = 60 r.p.s.,
W = reciprocating weight = 4.5 lb.,
T = period of oscillation = $\frac{1}{60}$ sec., and
x = 0.052/2 in. = $\frac{1}{2}$ distance traveled by reciprocating weight.

Since there is a mean load $D/3$ exerted on the wire test specimen, the total range of tension stress is:

Fiber stress

$$= [(+100 + 43) - (+100 - 43)] \div 0.00113$$

$$= +76{,}000 \text{ psi.}$$

where D = mean load = 300 lb.,
d = diameter of test specimen = 0.0379 in., and
a = area of test specimen = 0.00113 sq. in.

The stress in the specimen can be computed by the measured movement of the reciprocating weight and by direct measurement of strain in the wire. The calibration of the machine was carried out as follows: By means of a screw adjustment, the crank was offset to give equal increments in weight travel. By means of the micrometer microscope, sixteen readings were taken of the travel of this reciprocating weight; also the corresponding strains in a gage length of 8.77 in. These data, converted into stress values, were plotted by the method of least squares. The two straight-line relations, thus established, checked quite closely, which indicates that the machine functions according to the assumed principle. See Fig. 5. The points determined by strain measurements in the specimen are the more widely scattered. This may be explained by the fact that an error of 0.001 in. in measuring strain

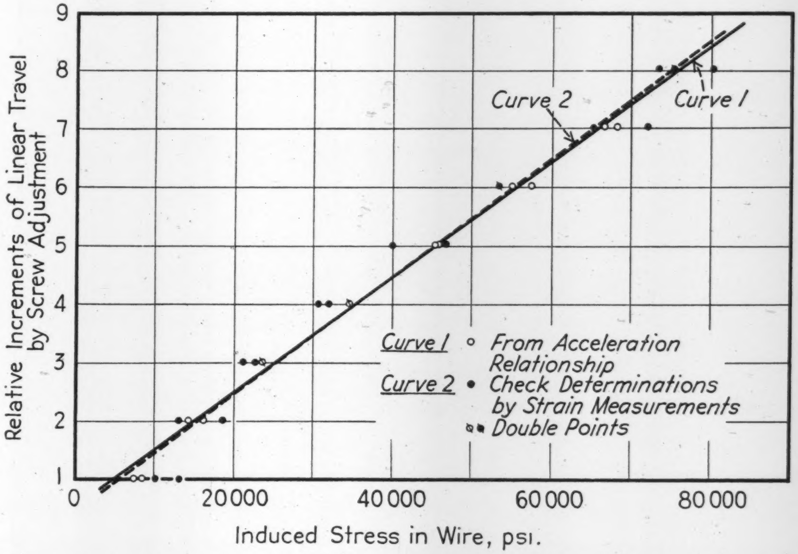

FIG. 5.—Pulsating Tension Calibration Data. Curves established by method of least squares.

is equivalent to an error in stress of 3400 psi. whereas a similar error in estimating the travel of the reciprocating weight is only equivalent to a stress of 1450 psi. In view of the performance of the machine, it is believed that the micrometer microscope, with special refinements in application, will prove to be sufficiently accurate for determining stress values.

The machine operates at the unusually high speed of 7200 stress-cycles per minute (3600 r.p.m.). The tendency of the test specimen assembly to assume a certain natural frequency is effectively balanced by two other similar assemblies which act as counterweights. The framework of the machine simply supports the reciprocating mechanism. The possibility of the driving disk developing a higher harmonic effect is unlikely since its period of natural frequency is vastly higher than that at which the machine operates.[3] If higher harmonic effects were appreciable, it might be expected to increase in intensity with increased load application. Such is not the case as evidenced by the straight-line relation between the crank-setting increments and the stress in the wire as determined by the reciprocating weight, Fig. 5.

The gripping of the ends of the wire specimens was accomplished by crimping on brass connectors. The ends of the test specimen were first polished to raise the fatigue limit. Then a $\frac{3}{8}$-in. threaded and a $\frac{1}{4}$-in. plain brass sleeve, each drilled out to the diameter of the wire, were slipped over the ends of the specimen leaving about $\frac{1}{2}$ in. of wire protruding. Each sleeve was then pressed on in a die under approximately 20,000 lb. pressure after which the extending ends of the wire were bent into loops and soldered. This gripping method proved effective since well over 80 per cent of the test specimens failed clear of the end connectors. See Figs. 6 and 9.

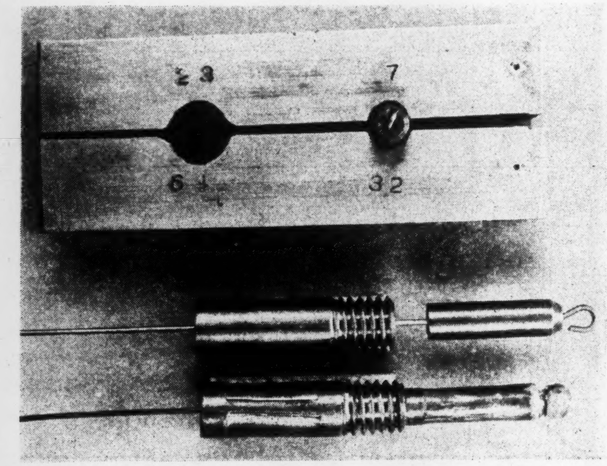

FIG. 6.—Show the Method of Gripping the Pulsating Tension Wire Test Specimens.

Test Results:

The materials tested in the machine were steel bead wire (automobile tire reinforcement). The loss in endurance properties was obtained on wire taken from worn automobile tires, thus exhibiting the practical application of the machine.

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[3] S. Timoshenko, "Vibration Problems in Engineering," D. Van Nostrand Co., New York, N. Y. (1937).

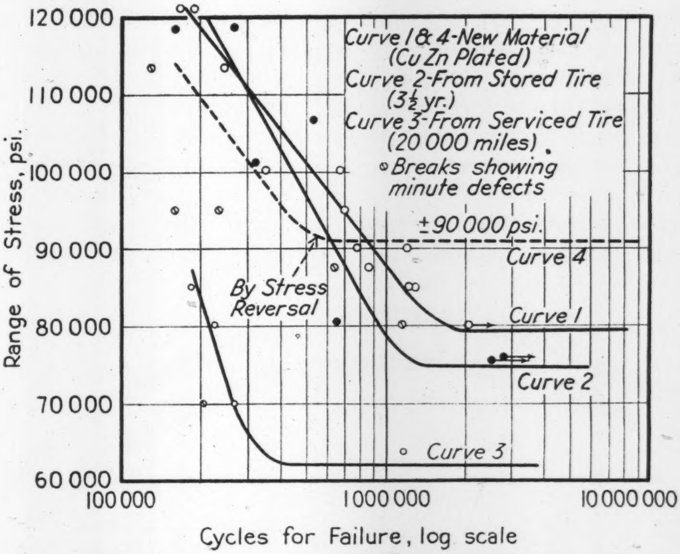

FIG. 7.—Pulsating Tension Tests on Bead Wire.

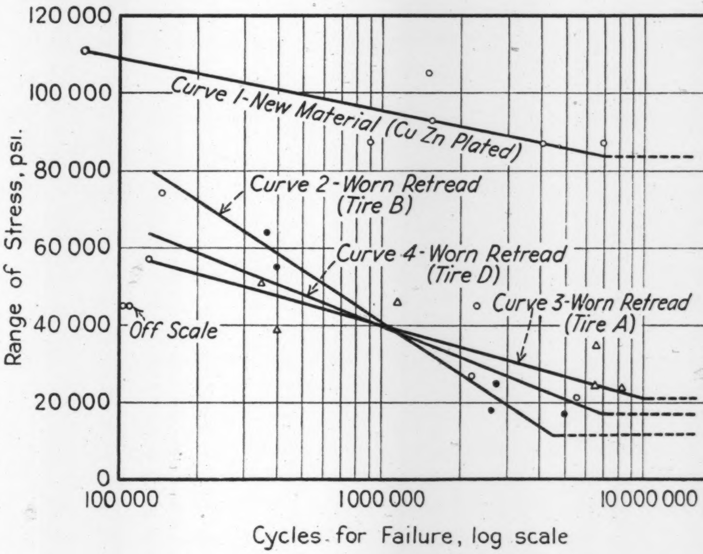

FIG. 8.—Pulsating Tension Tests on Bead Wire.

The results in Fig. 7 show a considerable scattering of points; however, some may be attributed to minute inhomogeneities in the wire materials. Curve 1 was obtained on unused bead wire and, for comparison, a curve was obtained by a stress reversal method,[4] see broken-line curve. The range of stress obtained by stress reversal is 12 per cent higher than that obtained by pulsating tension. Shelton and Swanger, at the National Bureau of Standards, investigated a series of bridge wires with the Haigh alternating stress machine. They found no effect on the endurance limit by varying the mean stress through a wide range; in no case was the range of stress obtained by pulsating tension higher than that obtained by stress reversal.[5] No direct comparison of results can be made on bead wire by means of the Haigh machine since the minimum measured force which it exerts is far above the breaking strength of this material.

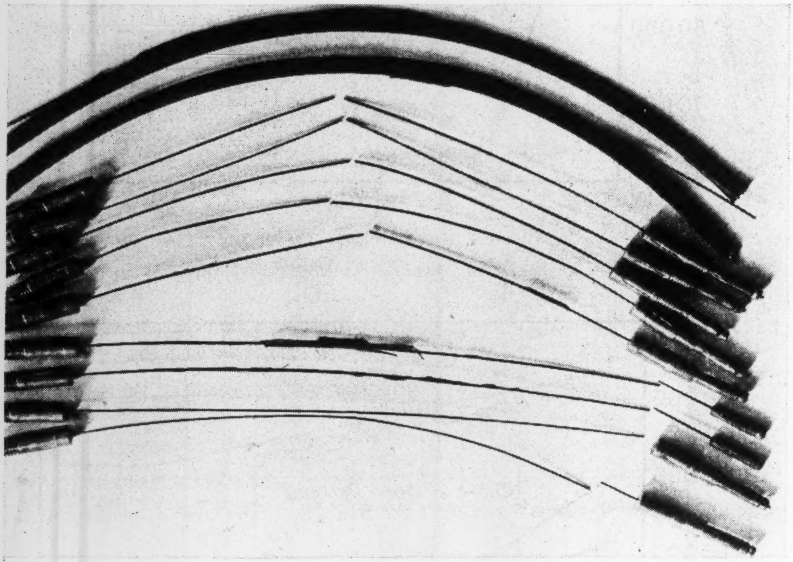

FIG. 9.—Broken Pulsating Tension Test Specimens.

Curve 2 of Fig. 7 shows data obtained on wire taken from a spare tire, never used in service, but subjected for 3.5 yr. to extremes in climatic temperatures and humid conditions. The tire was protected from actual contact with water by the tire carrier. The tests show no serious falling off in the endurance properties.

Curve 3 of Fig. 7 shows fatigue data obtained on wire taken from a tire which had been driven 20,000 miles, and with the tread worn thin. The trend of the curve is toward a lowered endurance value; however, the bead wire has outlasted the normal life of the tread.

The results shown in Fig. 8 were obtained on wire taken from worn retreaded tires as follows:

	Type of Car Used	Mileage	
		Original[a]	Retread
Tire A.........	Willys 77 ('33)	15 000	25 000
Tire B.........	Ford Coupe—Model A	20 000	11 000
Tire C[b]........	Ford Coupe—Model A	15 000	18 000
Tire D	Ford Coupe—Model A	15 000	18 000

[a] Estimated.
[b] See Fig. 11.

An examination of tires A and B showed that water had penetrated into the

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[4] J. N. Kenyon, "The Rotating-Wire Arc Fatigue Machine for Testing Small-Diameter Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 156 (1935).

[5] S. M. Shelton and W. H. Swanger, "Fatigue Properties of Steel Wire," *Journal of Research*, Nat. Bureau Standards, Vol. 14, No. 1, January, 1935, p. 17 (*Research Paper No. 754*).

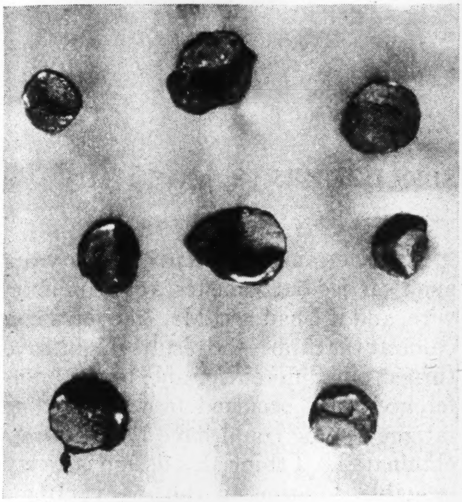

FIG. 10.—Pulsating Tension Fatigue Fractures.

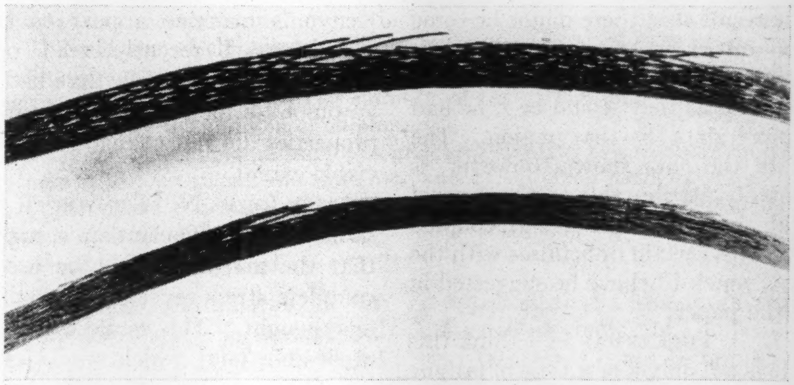

FIG. 11.—Steel Braid from Worn Retreaded Tire Showing Service Breaks.

bead, corroding the wire in spots. Pulsating tension tests, made on this wire gave ranges of stress of approximately 16,000 and 20,000 psi., respectively; obviously the wire is in the danger range. See curves 2 and 3 of Fig. 8. Broken test specimen fractures are shown in Figs. 9 and 10. Note that some of the fatigue breaks show evidence of deep pitting which is the incipient beginning of failure. Bead wire taken from the retreaded tire D was also badly corroded. The limiting range of pulsating stress was somewhat under 20,000 psi., although the scattering of points made the determination of this value difficult. See curve 4 of Fig. 8. Figure 11 shows steel bead wire braid taken from the retreaded tire C. This braid was broken in several places and seriously corroded. The deterioriated condition of the wire did not permit a determination of the endurance properties with the pulsating tension machine.

While no general conclusion can be drawn from these limited samplings of worn automobile tires, it is definitely believed that with this pulsating tension test method, further investigations may reveal important information.

Acknowledgment:

The author desires to acknowledge his indebtedness to J. K. Finch and W. J. Krefeld for their most cooperative attitude; to G. B. Karelitz for advice on the machine; to Colin G. Fink who called attention to the research possibilities offered by bead wire and who contributed most generously with constant advice to the study.

DISCUSSION

Mr. R. L. Templin.[1]—I think the author is to be congratulated on showing us another type of fatigue testing machine. I hope he will continue this work and extend his data somewhat. It would seem there is room to carry out some of the tests shown by the plotted data to a greater number of cycles. I do not believe there were any points indicated in the dotted portions of the curves which were drawn horizontally, with the result that there might be some question raised as to whether the apparent guesses as to endurance limits were as valid as they would be if he had some more data in that region. The scatter of the data shown, however, is undoubtedly attributable to the type of material that he was testing, coupled possibly with certain difficulties with the machine, which I believe he suggested in giving the paper.

Mr. L. B. Tuckerman.[2]—I think this machine is not only a new type of fatigue testing machine, but a very ingenious adaptation of a symmetrical three-phase harmonic motion system. What I am wondering is whether that cannot be applied to complete reversals of stress in a machine to handle other types of specimens so that we can get away from the nuisance of large fatigue testing machines. I do not know whether it can be done, but it would be highly desirable to have alternating tension and compression fatigue machines which used a constant torque.

Mr. Richard P. Seelig.[3]—Four years ago I carried out some tests on aluminum wire, and if I had had Mr. Kenyon's machine at that time, the results might have turned out differently, and many interferences that occurred in wire bending fatigue testing could have been entirely eliminated. I think this design is a very valuable addition to the many fatigue testing machines in use.

The tests carried out with Mr. Kenyon's machine appear to be very surprising. I recently read of some tests on machine parts that had run in automobiles for several years, and their properties did not change to any appreciable extent.

Mr. John N. Kenyon[4] (*Author's closure*).—Mr. Tuckerman's suggestion, that the machine might be used for a complete stress reversal, is food for serious thought. This would call for a type of flexible joint which would eliminate loose play at the zero or mean stresses. It is possible that valuable information could be obtained even on small size riveted joints and thereby gain time over the slower moving reciprocating machines.

I believe Mr. Templin's points are well taken. Some curves are incomplete, but we are building a new machine and expect to give more complete data sometime in the future. We certainly will continue the work.

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[1] Chief Engineer of Tests, Aluminum Co. of America, New Kensington, Pa.

[2] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

[3] Plant Manager, Powder Metallurgy, Inc., Long Island City, N. Y.

[4] Instructor in Civil Engineering, Columbia University, New York, N. Y.

THE CREEP STRENGTH OF 17 LOW-ALLOY STEELS AT 1000 F.

By R. F. Miller,[1] W. G. Benz[1] and W. E. Unverzagt[2]

Synopsis

The object of this investigation was to determine the effect of composition and heat treatment on the creep strength of the common low-alloy steels at 1000 F.

The creep strength of the carbon-molybdenum steels increases with increasing molybdenum content both in the normalized and in the normalized-and-tempered condition. In each composition the creep strength of the normalized material is higher than that of the normalized and tempered material. At 1000 F. the pearlitic structure resulting from the normalizing treatment is more unstable than the spheroidized structure resulting from normalizing and tempering.

The addition of chromium tends to lower the creep strength of carbon-molybdenum steel, but this decrease may be partially offset by increasing the molybdenum content. The stability of the carbon-molybdenum steels is increased by the addition of chromium.

Titanium or columbium improves the creep strength of chromium-molybdenum steels, but silicon and aluminum affect their creep strength adversely.

Conservative estimates of creep strength should be based on materials having a stable structure.

Three years ago, examination of the data for low-alloy steels in commercial use between 800 and 1100 F. revealed considerable differences in the reported creep strength of these steels. These discrepancies not only were a source of perplexity to engineers, but rendered uncertain any deductions as to the effect of an alloying element on creep strength. We considered it expedient, therefore, to redetermine the creep strength of some of these steels.

Since in general the relative order of creep strength of the low-alloy steels remains the same from 800 to 1100 F., it appeared that a broader picture of the influence of change of composition and heat treatment could be obtained in the same length of time by studying a wider variety of steels at a single temperature than by being restricted to a smaller number of steels at a series of temperatures. Accordingly, a temperature of 1000 F. was chosen for the comparison, and the same two stresses were used for all steels. The stresses, 10,000 and 15,000 psi., were selected because the available data indicated that they approximated the creep strength (stress corresponding to a creep rate of 0.000001 in. per inch per hour, or 1 per cent per 10,000 hr.) of these steels. In some cases, however, an additional stress was employed to permit estimation of the creep strength by interpolation rather than by extrapolation, so as to minimize the possible large error in any extrapolated value.

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[1] Research Laboratory, U. S. Steel Corp., Kearny, N. J.

[2] Metallographist, National Works, National Tube Co., McKeesport, Pa.

The results of this investigation, which are summarized in Table V, show closer agreement with the data published during the past year than had been anticipated. There are several reasons for this. First, the composition of the steels themselves is becoming more standardized. Second, while creep testing methods differ in detail in each laboratory, the major precautions to be observed are now so well recognized that substantially identical results on a given material are now obtained by practically all laboratories; this is shown by the cooperative tests on carbon steel at 850 F. sponsored by the Joint Research Committee on Effect of Temperature on the Properties of Metals of the A.S.T.M. and A.S.M.E.[3] The differences in reported creep strength now appear to be attributable rather to possible variation in grain size and prior heat treatment, and also to such minor differences in manufacturing practice and composition as still exist, than to differences in testing technique.

Composition of Steels:

The composition of the seventeen steels used in this investigation is given in Table I. Thirteen of the seventeen steels are standard compositions now in commercial use; four (steels A3, A4, C2, and C6) are of modified composition, included to show the effect of variation of the molybdenum content and of the addition of columbium. For comparative purposes, the steels have been divided into three groups.

TABLE I.—COMPOSITION OF STEEL.

Steel	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Chromium, per cent	Molybdenum, per cent	Other Elements, per cent
Group A								
A1	0.15	0.46	0.006	0.023	0.14	....	0.56	
A2	0.16	0.43	0.015	0.013	0.16	....	1.10	
A3	0.21	0.38	0.017	0.025	0.03	....	1.36	
A4	0.13	0.33	0.014	0.022	0.01	....	1.95	
A5	0.21	0.86	0.016	0.026	0.13	....	0.58	
Group B								
B1	0.12	0.47	0.008	0.007	0.22	2.06	0.50	
B2	0.10	0.34	0.012	0.007	0.12	2.26	1.06	
B3	0.14	0.43	0.011	0.008	0.84	1.25	0.54	
B4	0.17	0.52	0.012	0.014	0.57	1.78	0.78	
B5	0.12	0.48	0.010	0.011	1.57	3.26	0.54	
B6	0.08	0.32	0.010	0.011	1.43	3.02	0.54	Al 0.56
Group C								
C1	0.13	0.36	0.017	0.007	0.44	5.48	0.50	
C2	0.13	0.56	0.008	0.017	0.38	5.66	0.98	
C3	0.13	0.26	0.011	0.010	1.57	5.24	0.50	
C4	0.07	0.28	0.010	0.017	1.28	4.96	0.54	Al 0.58
C5	0.05	0.34	0.010	0.008	0.39	5.20	0.58	Ti 0.47
C6	0.09	0.42	0.012	0.011	0.35	5.00	0.55	Cb 0.59

Group A: Carbon-molybdenum steels, with varying molybdenum and manganese content.

Group B: 1.25 to 3.25 per cent chromium steels, with additions of molybdenum, silicon, and aluminum.

Group C: 5 per cent chromium steels, with additions of molybdenum, silicon, aluminum, titanium, and columbium.

Heat Treatment:

The heat treatment employed for each of these steels is shown in Table II. Because of the wide variation of composition, it was impossible to use the identical heat treatment throughout. For example, normalizing may be used for carbon-molybdenum steels without undue air hardening, but it will produce a marked increase in the hardness of chromium-molybdenum steels. Suitable heat treatments, representative of commercial practice, were therefore selected. The material was heat treated in the form of 1-in. rounds; after heat treatment, coupons were cut from the

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[3] C. L. Clark, "Report Covering Creep Tests on 0.35 per cent Carbon Steel (K20) at 850 F. Under a Stress of 7500 psi.," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 130 (1938).

TABLE II.—HEAT TREATMENTS.

Steel	Specimen	Heat Treatment
Group A		
A1–A4	a and b	Normalize 1650 F., 0.5 hr., air cool
A1–A4	c and d	Normalize 1650 F., 0.5 hr., air cool; temper 1300 F., 5 hr., air cool
A5	a and b	Normalize 1650 F., 0.5 hr., air cool; temper 1300 F., 5 hr., air cool
Group B		
B1	a and b	Normalize 1650 F., 1 hr., air cool; temper 1380 F., 4 hr., furnace cool
B2	a and b	Normalize 1650 F., 1 hr., air cool; temper 1380 F., 4 hr., furnace cool
B3	a	Anneal 1550 F., 1 hr., furnace cool
B3	b, c and d	Normalize 1650 F., 1 hr., air cool; temper 1380 F., 4 hr., furnace cool
B4	a and b	Normalize 1750 F., 1.5 hr., air cool; temper 1375 F., 2 hr., furnace cool
B5	a, b and c	Anneal 1600 F., 2 hr., furnace cool
B6	a, b and c	Normalize 1550 F., 3 hr., air cool
Group C		
C1	a and b	Anneal 1600 F., 2 hr., furnace cool
C1	c, d and e	Normalize 1600 F., 1 hr., air cool; temper 1380 F., 4 hr., air cool
C2	a and b	Normalize 1650 F., 0.5 hr., air cool; temper 1380 F., 4 hr., air cool
C3	a, b and c	Anneal 1600 F., 2 hr., furnace cool
C4	a, b and c	Normalize 1550 F., 3 hr., air cool
C5	a and b	Temper 1380 F., 4 hr., air cool
C6	a, b and c	Temper 1380 F., 4 hr., air cool
C6	d	Anneal 1600 F., 1 hr., furnace cool

TABLE III.—GRAIN SIZE.

Code	Heat Treatment, deg. Fahr.[a]	Grain Size Number, ASTM		
		Ferrite	Austenite	McQuaid-Ehn
A1	1650 N	8–9	7–*8*	6–7
	1650 N 1300 T	8–9	7–*8*	6–7
A2	1650 N	8–9	7–9	8–9
	1650 N 1300 T	8–9	7–9	8–9
A3	1650 N	9	Below A_3 temp.	2–3; 6–7
	1650 N 1300 T	9	Below A_3 temp.	2–3; 6–7
A4	1650 N	8–*9*	Below A_3 temp.	8–9
	1650 N 1300 T	8–*9*	Below A_3 temp.	8–9
A5	1650 N 1300 T	8–*9*	8	1–3; 7–8
B1	1650 N 1380 T	*8*–9	5	5–6
B2	1650 N 1380 T	10	7–9	7–8
B3	1550 An	9	Below A_3 temp.	4–6
	1650 N 1380 T	9	Below A_3 temp.	4–6
B4	1750 N 1375 T	9–10	4, 6–7	6–7
B5	1600 An	10	Below A_3 temp.	5–7
B6	1550 N	8	Below A_3 temp.	7–*8*
C1	1600 An	8–9	7–9	7–*8*
	1600 N 1380 T	*9*–11	7–9	7–*8*
C2	1650 N 1380 T	9–11	7–8	7–*8*
C3	1600 An	*10*–11	Below A_3 temp.	6–7
C4	1550 N	7–8	Below A_3 temp.	7–*8*
C5	Hot rolled and 1380 T	8–9	Below A_1 temp.	7–8
C6	Hot rolled and 1380 T	*7–8*; 11–12	Below A_1 temp.	8–*9*
C6	1600 An	10	Below A_3 temp.	8–*9*

[a] The following terms and abbreviations are employed:
N = normalize—air cool from top heat-treating temperature.
An = anneal—furnace cool from top heat-treating temperature.
T = temper—reheat below lower critical temperature.

TABLE IV.—TEST DATA, 1000 F. CREEP TESTS.

Creep data for individual time intervals, or other data not included in this table, will be supplied on request by the Research Laboratory of the U. S. Steel Corp.

Type of Steel	Specimen	Creep Stress, psi.	Duration of Creep Test, hr.	Creep Rate During Last 1000 hr., 0.000001 in. per inch per hour	Brinell Hardness		Modified Charpy Impact Strength, ft-lb.[a]	
					Before Creep Test	After Creep Test	Before Creep Test	After Creep Test
Group A								
A1	a	15 000	5000	5.16	131	134	39	34
	b	10 000	3200	0.52	131	134	39	38.5
	c	15 000	3100	17.1	116	128	40	37
	d	10 000	3000	1.02	116	114	40	40
A2	a	15 000	4150	1.65	146	179	39	33.5
	b	10 000	3100	0.43	146	174	39	38.5
	c	15 000	2900	2.13	143	137	42	42
	d	10 000	3300	0.39	143	140	42	41
A3	a	15 000	3000	0.88	167	197	26.5	24.5
	b	10 000	3000	0.36	167	201	26.5	25
	c	15 000	3000	1.17	163	163	29	31.5
	d	10 000	2000	0.52	163	174	29	32.5
A4	a	15 000	3000	0.47	152	212	42	18.5
	b	10 000	3000	0.17	152	223	42	6
	c	15 000	3000	0.78	173	167	38	38.5
	d	10 000	2000	0.22	173	163	38	39
A5	a	15 000	3000	2.34	152	156	38	36
	b	10 000	2400	0.56	152	152	38	33
Group B								
B1	a	15 000	1900	8.8	141	131	49	45
	b	10 000	3000	1.04	141	134	49	45
B2	a	15 000	3000	1.47	159	167	46	44
	b	10 000	3000	0.54	159	163	46	42.5
B3	a	22 000	2100	24.2	156	159	41.5	21
	b	15 000	1300	33.6	146	—	36	—
	c	10 000	3000	3.8	146	152	36	33
	d	7 000	2300	0.59	146	137	36	35
B4	a	15 000	3000	2.9	181	163	40.5	27
	b	10 000	3000	0.71	181	179	40.5	26
B5	a	15 000	800[b]	87.6	170	170	31	26
	b	10 000	3000	4.12	170	170	31	20
	c	7 500	3000	0.67	170	167	31	16.5
B6	a	15 000	200[b]	98.1	163	159	21 (2)	27.5
	b	10 000	1000[b]	5.7	163	170	21 (2)	26.5
	c	6 500	3000	0.4	163	156	21 (2)	25.5 (1.5)
Group C								
C1	a	15 000	200[b]	94.0	136	134	38	31.5
	b	10 000	2000	2.0	136	146	38	31.5
	c	15 000	1000[b]	13.9	191	174	50	45
	d	10 000	3000	3.56	191	172	50	43
	e	7 000	3000	1.19	191	179	50	43
C2	a	15 000	1000[b]	7.9	202	174	39	33
	b	10 000	2200	0.72	202	212	39	35
C3	a	15 000	500[b]	84.3	170	170	38	35.5
	b	10 000	2200	4.56	170	167	38	23.5
	c	7 500	3000	0.30	170	170	38	28
C4	a	15 000	200[b]	163	154	163	51	46.5
	b	10 000	1700	18.8	154	149	51	40 (1.5)
	c	5 500	2200	1.2	154	152	51	41
C5	a	15 000	1500	32.3	149	134	52	44
	b	10 000	2200	0.2	149	126	52	43
C6	a	15 000	2000	0.28	169	156	49	36
	b	10 000	2000	0.28	169	149	49	43
	c	20 000	1500	12.8	169	163	49	47.5
	d	15 000	900	41.7	140	140	46.5	38

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[a] Values are averages of two tests. When results are in wide disagreement the second test value is indicated in parenthesis.

[b] The creep rate exceeded the capacity of the apparatus; other tests were run for longer times under lower stresses to supply data necessary for determination of stress for desired creep rate.

ends of the 8-in. bars for determination of microstructure, and the remainder was machined to form the creep specimen.

In order to study further the effect of different heat treatments on creep strength, discussed in a previous paper,[4] two separate heat treatments were employed for group A and for certain of the steels of groups B and C.

TABLE V.—CREEP STRENGTH AT 1000 F.

Code	Heat Treatment, deg. Fahr.	Stress for Creep Rate of 0.000001 in. per inch per hour, psi.	
		Kearny	Other Laboratories
A1	1650 N	11 500	12 400 (B & W) 10 800 (Timken)
	1650 N 1300 T	10 000	
A2	1650 N	13 300	11 600 (B & W)
	1650 N 1300 T	12 800	
A3	1650 N	15 800	
	1650 N 1300 T	14 300	
A4	1650 N	18 800 (extrapolated)	
	1650 N 1300 T	15 900	
A5	1650 N 1300 T	12 000	
B1	1650 N 1380 T	10 000	8 900 (Timken) 11 000 (B & W)
B2	1650 N 1380 T	13 300[a]	11 000 (Timken) 16 350 (B & W)
B3	1650 N 1380 T	7 800[a]	22 000 (Timken)
B4	1750 N 1375 T	11 300	
B5	1600 An	8 500[a]	8 700 (Timken)
B6	1550 N	7 800[a]	8 400 (Timken)
C1	1600 An	9 100[a]	9 200 (B & W) 10 100 (Timken) 9 725 (Climax)
	1600 N 1380 T	6 600[a]	
C2	1650 N 1380 T	10 800[a]	10 000 (Climax)
C3	1600 An	8 800[a]	8 700 (Timken)
C4	1550 N	5 200[a]	7 600 (Timken)
C5	1380 T	11 500	13 000 (B & W)
C6	1380 T	16 400	

[a] Stable structure.

For the purpose of facilitating correlation of the data, the grain size of the steels was observed, and the results are listed in Table III. The *ferrite* grains are those existing in the steel at room temperature after the final heat treatment and before creep testing, determined on the coupons cut from the heat-treated bars before machining. All of the steels are fine-grained in the ferritic condition. The *austenite* grains are those produced at the maximum heat treating temperature for each steel. The austenite grain size was determined on separate coupons of each steel, 1 in. in diameter by 0.25 in. thick, which were quenched in brine from the heat-treating temperature and then slightly tempered to improve definition of the austenite grains. It will be noted that the ferrite grain size is proportionate to and slightly smaller than the austenite grain size. The *McQuaid-Ehn* grain size is the size of the *austenite* grains produced in the carburized surface layer of separate coupons of each steel (1 in. in diameter by 0.5 in. thick) after carburizing for 8 hr. at 1700 F. followed by furnace cooling. Therefore, the McQuaid-Ehn grain size is not the same as the austenite grain size in the steel as heat treated for creep testing. A complete definition of these terms for grain size and the method of their determination is contained in a recent paper by Vilella.[5]

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[4] R. F. Miller, R. F. Campbell, R. H. Aborn and E. C. Wright, "Influence of Heat Treatment on Creep of Carbon-Molybdenum and Chromium-Molybdenum-Silicon Steel," *Transactions*, Am. Soc. Metals, Vol. 26, p. 81 (1938).

No change of ferrite grain size was noted in any of the specimens after exposure to the creep test.

Creep Strength at 1000 F.:

The creep testing apparatus employed in these tests has been described in previous publications.[4,6] During the course of this testing program, an improved creep measuring microscope was developed and installed, which has a sensitivity of ± 0.00002 in. and a maximum precision of ± 0.00001 in. per inch (± 0.001 per cent). The specimens have a diameter of 0.505 in. and a gage length of 2.3 in.

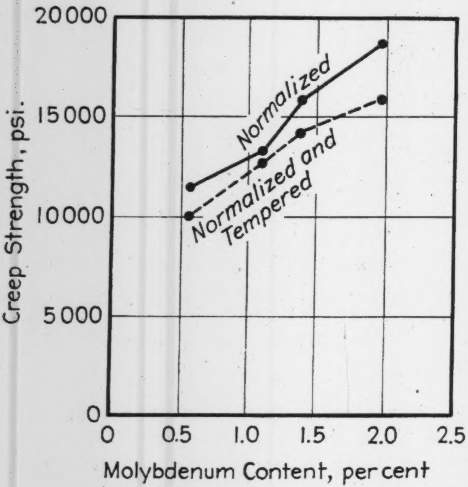

FIG. 1.—Effect of Molybdenum Content on Creep Strength of Carbon-Molybdenum Steels at 1000 F. (Group A).

The temperature variation over the gage length of the specimen is ±1 F. and the control is ±1.5 F. The irregularities in the extension measurements and hence in the creep curves were of about the same order of magnitude as the changes in length produced by the ±1.5 F. fluctuation of the temperature in the furnace. All creep tests were carried out in conformity with the A.S.T.M. Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 - 38 T).[7]

The stress employed for each individual specimen, the duration of the test, and the creep rate during the last 1000 hr. of each test are listed in Table IV. From these data, the creep strength (stress for a creep rate of 0.000001 in. per inch per hour) of each steel has been calculated, as is shown in Table V.

The creep strength of the carbon-molybdenum steels increases with increasing molybdenum content, both in the normalized and in the normalized-and-tempered condition, as shown in Fig. 1. For each composition, the creep strength of the normalized material is higher than that of the normalized-and-tempered material. However, the normalized material is less stable than the normalized-and-tempered material, and the creep strength of both may become the same after a prolonged exposure to a temperature of 1000 F. The topic of stability of microstructure is discussed more fully in the next section. Increase of carbon and manganese content appears to cause a slight increase in the creep strength of normalized-and-tempered carbon, 0.5 per cent molybdenum steel, as seen from a comparison of steels A1 and A5, Table V.

Figure 2 shows graphically the effect on creep strength of change of composition of similarly heat-treated chromium-molybdenum steels. The creep strength of normalized-and-tempered steel containing 2 or 5 per cent chromium is enhanced by raising the molybdenum content from 0.5 to 1 per cent; but it is lowered by raising the chromium from 2 to 5 per cent. A similar lowering is observed when the chromium content of normalized chromium-molybdenum-silicon-aluminum steel is raised from 3 to 5 per cent. On the other hand, increase of chromium from 3 to 5 per cent resulted in little change of the creep strength of annealed steel containing 0.5 per cent molybdenum and 1.5 per cent silicon.

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[5] J. R. Vilella, "The Grain Size of Steel," *Mechanical Engineering*, Vol. 62, No. 4, April, 1940, pp. 293–307.

[6] Richard F. Miller, "The Strength of Metals at Elevated Temperatures," *Mechanical Engineering*, Vol. 61, No. 8, August, 1939, p. 589.

[7] 1939 Book of A.S.T.M. Standards, Part I, p. 1261.

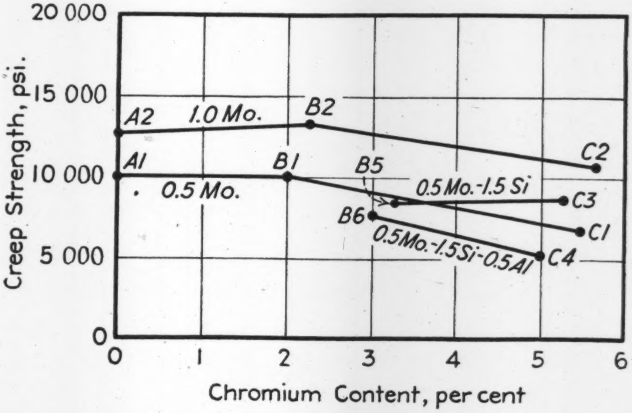


FIG. 2.—Effect of Chromium Content on Creep Strength of Chromium-Molybdenum Steels at 1000 F. (Groups B and C).

Steels B3 and B4 both contain chromium, molybdenum, and silicon, were similarly heat treated, and have a similar grain size. The slightly higher alloy content of B4 results in a considerably more creep-resistant material. A possible reason for this enhanced creep strength is that this small change in composition markedly increases the air-hardening propensities of this type of material; in other words, the normalizing and tempering heat treatment yields a different microstructure in the two steels. In steel B4, the carbide particles are found to be fine and uniformly distributed, while in steel B3 they have coalesced at the grain boundaries where they are presumably less effective in strengthening the material.

The creep strength of steel B3 was found to be 7800 psi. at 1000 F., considerably lower than that, 22,000 psi., reported by another laboratory. Consequently, a sample of steel B3 was heat treated (annealed) in the manner reported to have been employed in obtaining the higher creep strength, and this sample was tested at a stress of 22,000 psi. The creep rate during the last 1000 hr. of this test was 0.000024 in. per inch per hour, a rate so rapid that the lower value was confirmed as being a safer estimate of its creep strength.

Remarkably good results were obtained with steels C5 and C6 in the hot-rolled and tempered condition. Steel C5 shows a creep strength of 11,500 psi. as compared to 6600 psi. for the same steel (C1) in the normalized-and-tempered condition without titanium. Steel C6 has a creep strength of 16,400 psi., the best of any steel tested except the normalized carbon-molybdenum steel A4, which has a creep strength of 18,800 psi. The creep rate under a stress of 15,000 psi. at 1000 F., of steel C6 in the annealed condition was ob-

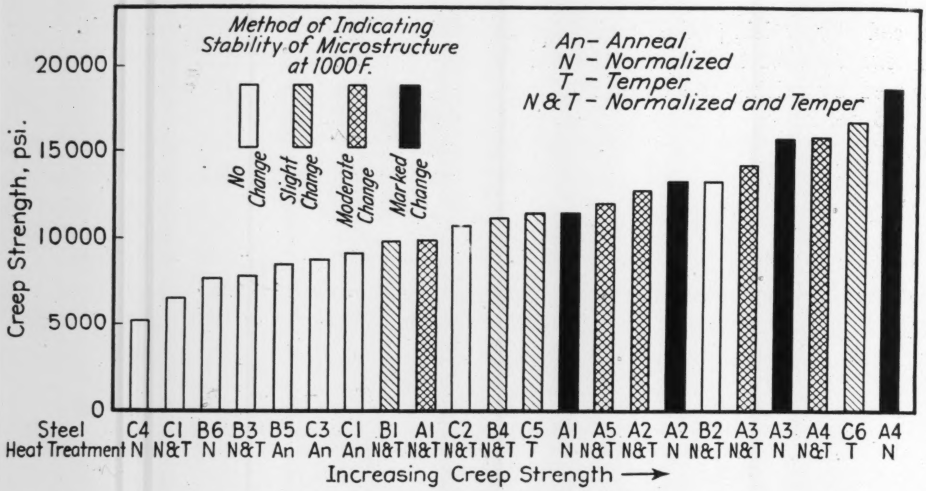
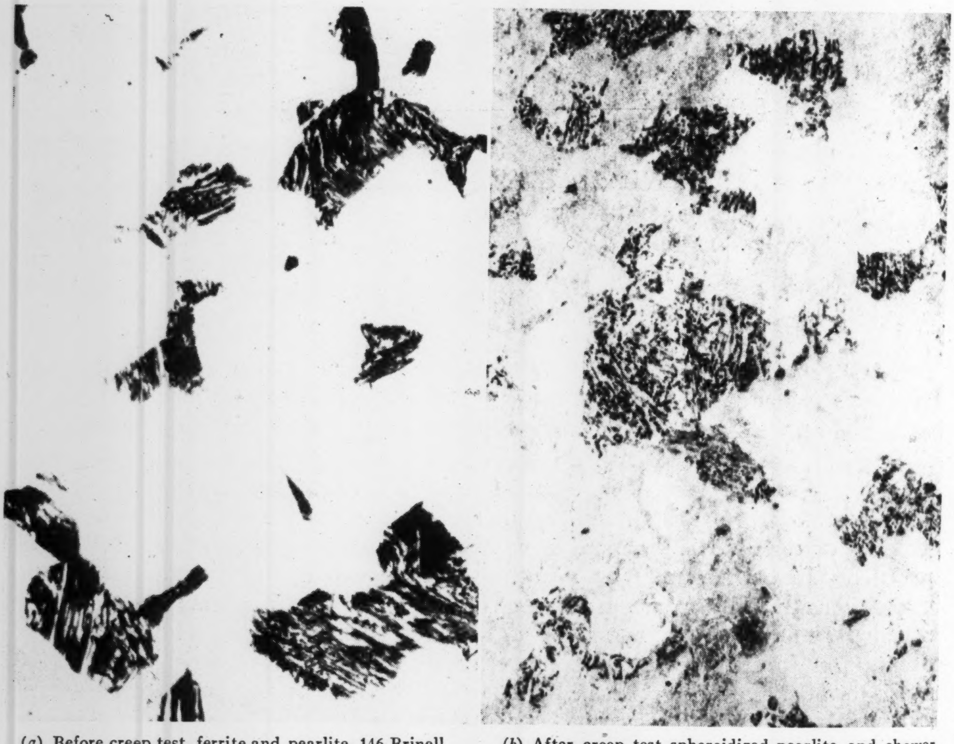


FIG. 3.—Steels Plotted in Order of Increasing Creep Strength Showing Relative Stability of Microstructure During Creep Test at 1000 F.



(*a*) Before creep test, ferrite and pearlite, 146 Brinell.

(*b*) After creep test spheroidized pearlite and shower precipitate of complex molybdenum carbide in ferrite, 179 Brinell.

FIG. 4.—Change of Microstructure of Normalized Steel A2 After 3000 hr. Exposure to Creep Test at 1000 F. (× 1000).

served to be 0.000042 in. per inch per hour, but in the hot-rolled and tempered condition to be only 0.00000028 in. per inch per hour; this is about 150 times less rapid. Although the creep strength of steel C6 is lower in the annealed than in the hot-rolled and tempered condition, it is still above that of the same steel without columbium. This is demonstrated by a comparison of the creep rates of steels C1 (without columbium) and C6 (with columbium) in the annealed condition under a stress of 15,000 psi. (0.000094 and 0.000042 in. per inch per hour, respectively).

In general it may be said that, excepting the titanium- and columbium-bearing steels, the creep strength of the steels in group C is somewhat lower than that of the steels in group B, while that of group B is somewhat lower than that of group A.

Stability of Microstructure:

The microstructure of all of the specimens was examined, both before and after creep testing, and the observations regarding the relative stability of the microstructures of these steels at 1000 F. are summarized in Fig. 3. The change of microstructure observed in a given steel with a given heat treatment seemed to be unaffected by the stress, and to depend solely on the duration of its sojourn at 1000 F.

The most pronounced change of microstructure was found in the normalized carbon-molybdenum steels of group A. An example of this change of microstructure due to exposure to the creep test is shown in Fig. 4. There was a moderate change in the normalized-and-tempered carbon-molybdenum steels, and in the annealed steel B3. In the other chromium-molybdenum steels of groups B and C there was a very slight change, or no visible change at all. It is thought that the superior stability of the steels of groups B and C as compared to those in group A may be ascribed to the chromium content, because both in the pearlitic and in the spheroidized condition, additions of chromium increased the stability of the microstructure.

Structures that are unstable at 1000 F. may be so altered by heating for a few hours at about 1300 F. that they will remain essentially unchanged however long they are exposed to the lower temperature of 1000 F. In other words, the same amount of structural change may be brought about in a few hours at the higher temperature that would have taken thousands of hours at the lower testing temperature.

Examination of Fig. 3 shows that in general the steels with the highest creep strength are the ones with the least stability. Also, that when two heat treatments are used for the same steel, the more stable structure has the lower creep strength. If the creep tests had been run long enough at 1000 F. to permit the same change of structure which was produced by the subcritical heating (tempering) before creep testing, it is probable that the high creep strength data would not have been obtained. In this case, even 3000 hr. was not long enough to disclose the magnitude of the decrease of creep strength which would be encountered.

The design engineer would be well advised to inquire whether the microstructure of a steel he is considering for a given application is stable at the operating temperature, since the creep strength quoted for a given steel may represent a transitory condition and over a period of time the creep strength of the material may decrease considerably. Conservative estimates of the creep strength of a given material should be made on the basis of a structure known to be stable at the service temperature.

Impact Strength and Hardness:

The impact strength of the steels, as determined before and after creep testing, is listed in Table IV. In order to determine the impact strength of the steels before creep testing, specimens of each steel 1 in. in diameter by 4 in. long were heat treated in the same manner as the creep specimens, and two duplicate Charpy keyhole impact specimens were then machined from each bar. Since the creep specimens from which the keyhole Charpy impact specimens were machined after creep testing had a diameter of 0.505 in., it was necessary to employ the substandard, two-thirds size specimen (hereafter called the "modified" Charpy impact specimen), which has a cross-section measuring $6\frac{2}{3}$ by 10 mm. instead of 10 by 10 mm.

The only significant effect on impact strength which could be attributed to composition occurred in steels B6 and C4. The initial heat treatment of these materials resulted in exceedingly coarse ferrite grains (A.S.T.M. No. 0 to 2) in the outer portions of the bar stock. Even though an attempt was made to machine off this coarse-grained skin in the preparation of the creep specimens, it is believed that some of this coarse-grained material was included in two of the impact specimens of steel B6 and in one of the impact specimens of steel C4, resulting in the abnormally low modified Charpy impact values of 1.5 and 2.0 ft-lb. No consistent trend in impact values could be attributed to differences in the heat treatment of individual steels.

Exposure of the steels to a temperature of 1000 F. for 3000 hr. caused little if any change in the impact strength of the carbon-molybdenum steels in group A. Steel A4 was an exception; in the normalized condition, the modified Charpy impact strength decreased from 42.0 ft-lb. in the initial material to 18.5 and 6.0 ft-lb. after creep testing under 15,000 and 10,000 psi., respectively. This may be associated with the corresponding increase in hardness from 152 Brinell to 212 and 223 Brinell. The chromium-molybdenum steels in groups B and C show a tendency toward lower impact strength after creep testing, and the decrease is more pronounced in the steels containing silicon.

The hardness of the steels was determined before creep testing on the microstructure coupons cut from the heat-treated bars before machining the creep specimens. The hardness of the specimens after creep testing was measured on the broken impact specimens which were machined from the creep specimens. The values are listed in Table IV. The hardness of the normalized carbon-molybdenum steels (group A) increases with increasing molybdenum content except in steel A4, which contains only 0.13 per cent carbon. A marked increase in the hardness of these normalized carbon-molybdenum steels

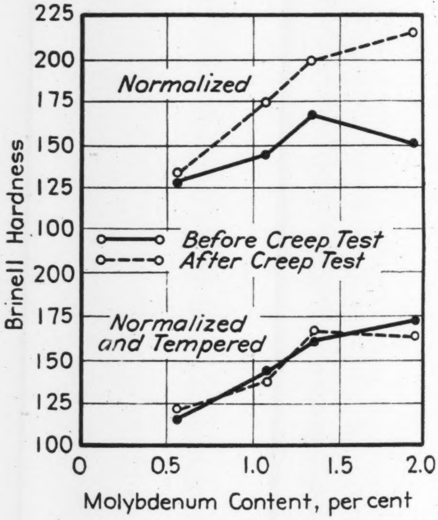


FIG. 5.—Effect of Molybdenum Content on Hardness of Carbon-Molybdenum Steels Before and After Creep Testing at 1000 F. (Group A).

occurs during creep testing as shown graphically in Fig. 5. It will also be seen that the hardness of the normalized and tempered carbon-molybdenum steels increases with increasing molybdenum content, but that in this condition there is very little change after creep testing.

In the chromium-molybdenum steels, the only significant trend attributable to change of composition is that an increase of molybdenum content in steels containing 2 or 5 per cent chromium results in an increase in hardness, both before and after creep testing.

After exposure to the creep test at 1000 F., the chromium-molybdenum steels of groups B and C show either a slight decrease of hardness, or no change.

Summary:

The creep strength (stress for a creep rate of 0.000001 in. per inch per hour) of the carbon-molybdenum steels increases with increasing molybdenum content, both in the normalized and in the normalized-and-tempered condition (Fig. 1). In each composition, the creep strength of the normalized material is higher than that of the normalized-and-tempered material. However, the pearlitic structure resulting from the normalizing treatment is unstable at 1000 F., and the lower creep strength of the more stable normalized and tempered materials represents a more conservative estimate of their creep strength. Increase of manganese and carbon content appears to increase slightly the creep strength of normalized-and-tempered carbon, 0.5 per cent molybdenum steel.

The addition of chromium tends to lower the creep strength of carbon-molybdenum steels (Fig. 2). However, this decrease may be partially offset by increase in the molybdenum content. Chromium increases the stability of carbon-molybdenum steels. The steel having the highest creep strength and at the same time a stable microstructure is B2.

The creep strength of the chromium-molybdenum steels is affected adversely by the addition of silicon and aluminum.

Addition of titanium or columbium to chromium-molybdenum steel improves its creep strength in the hot-rolled and tempered condition. However, a single test on the chromium-molybdenum-columbium steel in the annealed condition indicates that this improvement may depend on an initial heat treatment yielding a structure whose permanence is unknown.

A list of published values for the creep strength of some of the steels included in this program may be found in Table V. The values agree in general with those found in the present investigation, except in the case of steel B3. The improvement in creep strength brought about in chromium-bearing steels by increase in the molybdenum content is confirmed by recent work of other investigators.[8] It is our conclusion that if the analyses of the steels alluded to in the published data were within the range given by the A.S.T.M. specifications, and if the heat treatment and grain size of the steels were known and reproducible, it would now be possible to correlate creep test data obtained in different laboratories within 2000 psi.

Acknowledgment:

The authors wish to express their appreciation for the assistance of R. F. Campbell, N. A. Mathews, and others of the staff of the Research Laboratory in Kearny, and to E. C. Wright and the National Tube Co. for providing the material and advice concerning the heat treatment of the steels.

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[8] *The Moly Matrix*, Vol. 6, No. 7, July, 1939; Vol. 6, No. 9 September 1939.

DISCUSSION

Mr. Ernest L. Robinson.[1]— This paper represents a welcome volume of creep test work. Toward the end the authors caution designing engineers to avoid the use of short-time creep data on unstable materials. The writer wishes to express his agreement with this statement and add a word as to its importance.

It seems fairly plain that long-time information is necessary on any material which undergoes gradual change in its metallurgical characteristics during its period of service. However, the important thing is its long-time strength. While the materials presented here for the most part deteriorate with time, some other materials are known to improve. Furthermore, a material which deteriorates with time may be so outstandingly better at the beginning as to give it greater strength throughout the intended periods of service as compared with a weaker material less subject to change. Only long-time tests can provide the answer to these questions.

All the materials tested by the authors are, according to them, very fine-grained. It seems very likely that a coarser-grain treatment would have given improved creep strength at 1000 F. However, it is uncertain as to just what is meant by grain size without the benefit of the actual micrographs. Furthermore, all the materials dealt with are alloy steels, whereas the Vilella article referred to dealt only with plain carbon steel and specifically avoided the discussion of the grain size of alloy steels.

If each specific material had been given the benefit of its best treatment, the conclusions arrived at as to the relative creep strengths might have been quite different. There is good reason to believe that in many of these compositions the heat treatment is more important than the precise percentage of alloy.

Another matter that is very important is the fact that the authors have used loadings much too high for ordinary working stresses, and consequently it is uncertain whether or not they may be reliable as guides for determining working stresses. The A.S.T.M.-A.S.M.E. Creep Data volume expresses creep in millionths of the length per hour. The authors of the present paper have chosen a unit creep rate of this magnitude for the determination of their nominal strength. In the opinion of the writer a creep rate 100 times slower would be more appropriate. Thus, the nominal strengths given in the paper correspond to what is ordinarily spoken of as a rate of creep of 10 per cent per 100,000 hr., and this figure is likely to be well above the stress which will cause rupture at the end of 100,000 hr.

While the various test points are given in the paper, it is necessary for the reader to plot the information in order to find out what the indicated creep strengths would be for a rate of 1 per cent per 100,000 hr. When this is done, it is noted that in every case extrapolation is required to determine this value. There is good reason to believe that extrapolation to still lower values is not well justified.

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[1] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

However, comparison on the basis of a creep rate of 1 per cent per 100,000 hr. is illuminating. Considering the carbon molybdenum steels in group A, the weakest of all is material A3 containing 1.36 per cent of molybdenum which was one of the best as presented by the authors. On the other hand, their poorest material, A1 containing 0.56 per cent of molybdenum, becomes one of the best when rated with reference to a creep rate of 1 per cent per 100,000 hr.

Considering group B, the outstanding differences in creep strength for a 10 per cent rate disappear when the nominal strength is based upon rate of creep of 1 per cent per 100,000 hr. The whole lot ranges in value from 5000 for B2 and B3 to 6300 for B1. When the materials which show such outstandingly high strength for a 10 per cent rate fall off so rapidly when compared on the basis of a 1 per cent rate, it seems likely that they might be found particularly weak when still lower rates of creep are required. This just goes to show how difficult it is to select material of good quality when the tests are conducted under conditions departing widely from service loadings.

In group C materials, containing 5 per cent of chromium and 0.5 per cent of molybdenum, with additions, C6 is described as outstandingly strong. In order to arrive at the figure given in Table V it is necessary to consider the favorable specimen *a* in the tempered condition and to neglect the specimen *b* in which the resulting creep rate with a stress of 10,000 psi. was equal to that with specimen *a* at 15,000 psi. Doubtless the authors have a good reason to discount specimen *b* but it seems necessary to point out that if the line on the log-log plot is drawn in the more conservative location, through specimen *b*, the corresponding entry in Table V would be 12,500 psi. and with the steeper slope the material would not be so good as C5 on the basis of 1 per cent per 100,000 hr.

The writer recommends that in future the authors conduct their tests at loadings more nearly approaching what would be reasonable for service.

MR. M. B. HIGGINS.[2]—I was pleased that the results were presented by converting the determinations to a rate of creep of 1 per cent in 10,000 hr. As a rule creep data are presented to the engineer by giving percentage of elongation, but are not converted down to the common basis of 10,000 or 100,000 hr. As Mr. Robinson stated, there may be some question as to the method used by engineers in applying these figures properly. I have one question, and that as a matter of information: Is it safe to use the rate of creep and elongation over the last 1000 hr. only when converting laboratory determinations to rates per fixed time?

THE CHAIRMAN (*Mr. H. J. French*[3]).—I should appreciate having the authors give their definition of "stable structures." Their use of the term "stability" may differ somewhat from that which has been current in recent years.

The old idea with respect to high-temperature creep properties revolved about a spheroidized carbide, but I suspect that Mr. Miller and his associates use the term "stability" to define an unchanging structure within the time intervals of exposure, whether or not the carbides become spheroidized.

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[2] The Texas Company, New York, N. Y.

[3] In Charge, Alloy Steel and Iron Development, The International Nickel Co., Inc., New York, N. Y.

This point may be important in interpretation of the authors' results.

MR. A. J. HERZIG.[4]—I should like to ask the authors this question: In view of the fact that there is so much interest in this precipitation problem, what work has been done to identify the precipitate which is revealed in their metallographic examinations?

MR. J. M. LESSELLS.[5]—Reference might well be made to the work of Inglis[6] who compared impact values before and after subjecting the material to high-temperature conditions. A comparison of the two sets of data might be of interest.

MR. C. L. CLARK[7] (*by letter*).—It is gratifying to see, from Table V of the paper, the close agreement in the creep results from the different laboratories for the majority of steels considered. This represents an entirely different situation from that which existed a few years ago. Furthermore, if more complete information were available with respect to all of the steels, and especially with respect to their microstructures in the as-tested condition, it is believed that the variations which are present in certain of the results could be readily explained.

As an example, steel B3 may be considered. This is the one for which the authors obtained a stress value of 7800 psi. as compared to our previously reported stress of 22,000 psi. The ferritic grain size of the authors' specimens is reported in Table III as being 9, while the McQuaid-Ehn grain size is 4–6. The authors submitted to us specimens of this same heat in supposedly the same condition of heat treatment, that is, normalized from 1650 F. followed by a 4-hr. temper at 1380 F., and the microstructure, given in the accompanying Fig. 1, shows the predominating ferritic grain size to be 7. Our creep stress for these particular specimens, was 10,000 psi., as compared to the authors' 7800 psi. This difference of 2200 psi. is believed to be mainly due to the difference in ferritic grain size, 7 as compared to 9.

The structure of the specimen which possessed the creep strength of 22,000 psi. is shown in the accompanying Fig. 2. This structure was obtained by finish hot working at a sufficiently high temperature to produce a coarse grain followed by cooling (in air) at a rate to preserve the Widmanstaetten type structure. The steel was then annealed, as indicated, from 1550 F. which, for the particular composition considered, was just below the upper critical range. The structure of this same steel after annealing from 1650 F. is shown in the accompanying Fig. 3.

The superior creep resistance of this steel in the annealed from 1550 F. condition is due not only to its coarse grain but also to its Widmanstaetten type structure and, of the two, this latter is believed to be the most effective. For this steel, at least, this structure possesses good stability at 1000 F., for the room temperature tension, impact, and hardness characteristics of the completed 1000 F. creep specimens were practically identical to those of the original steel.

In reviewing the results for certain of the other steels, a question arises with respect to why certain of the heat treatments were employed and as to the procedure used for determining stability at 1000 F. For example, the tempering temperature used for B4 was slightly lower than that for the lower alloy steels of this same general type, and the time of tempering was 2 hr. rather than 4. This treatment imparted to steel B4 a Brinell hardness of 180 as compared to 140 to 160 for steels B1, B2, and B3. Likewise the structure of Steel B4 is shown, in Fig. 3 of the paper, to undergo only a slight change after the creep tests at 1000 F. yet the impact resistance has been reduced nearly in half and the hardness

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[4] Chief Metallurgist, Climax Molybdenum Co. of Michigan, Inc., Detroit, Mich.

[5] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[6] N. P. Inglis, "The Relationship Between Mechanical Tests of Materials and Their Suitability for Specific Working Conditions," *Transactions*, North East Coast Inst. Engrs. and Shipbuilders, Vol. 53, 1936-1937, pp. 101-136.

[7] Research Engineer, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

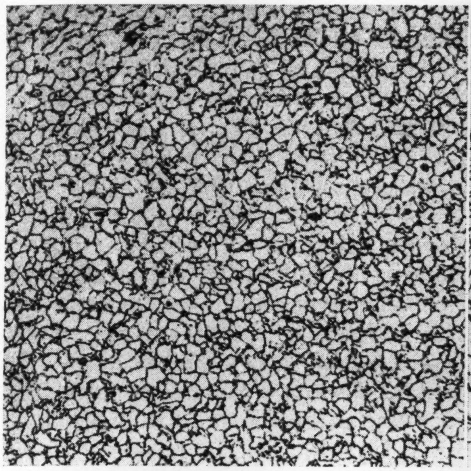

FIG. 1.—0.14 per cent Carbon, 0.84 per cent Silicon, 1.25 per cent Chromium, 0.54 per cent Molybdenum Steel.

Normalized at 1650 F., tempered at 1380 F. (4 hr.). 1000 F. creep strength; 10,000 lb. for 0.000001 per cent per hour.

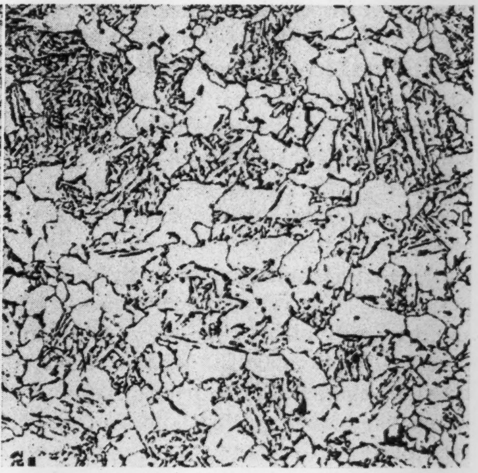

FIG. 2.—0.07 per cent Carbon, 0.72 per cent Silicon, 1.25 per cent Chromium, 0.54 per cent Molybdenum Steel.

Annealed at 1550 F. 1000 F. creep strength; 22,000 lb. for 0.000001 per cent per hour.

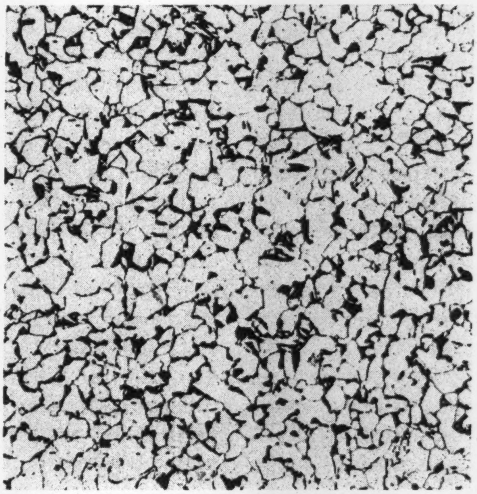

FIG. 3.—Same Steel as in Fig. 2 but Annealed from 1650 F. Rather than 1550 F.

of one specimen decreased 18 points. On the other hand, certain of the steels indicated in Fig. 3 as undergoing marked changes do not show as pronounced changes in impact resistance or in hardness.

With respect to the compositions of certain of the modified 4 to 6 per cent chromium-molybdenum type steels which were considered, Table I, the titanium content is slightly greater than nine times the carbon content, while the columbium content is somewhat greater than six times the carbon content. This is just the reverse of the manner in which these two alloying elements are usually added and I wonder whether the authors have information which would show that their procedure, rather than that generally used, is preferable.

MESSRS. R. F. MILLER,[8] W. G. BENZ[8] AND W. E. UNVERZAGT[9] (*Authors' closure by letter*).—The authors wish to express their appreciation for these comments and suggestions.

In regard to the discussion presented by Mr. Robinson, it may be that a coarser grain size would have resulted in higher creep strength in these alloys. However, we feel that the whole subject of the influence of grain size on creep strength has been somewhat oversimplified in the past. Evidence is accumulating that grain size in itself does not control the creep strength of a given steel; the distribution of the phases within the individual grains may cause as much change of creep strength as change of grain size or change of composition. Furthermore, unless the manufacturing practice and actual composition of a given steel are altered, it may be impossible to increase the grain size without using a heat treatment which is commerically impractical.

While we would like to know the best heat treatment for each of the steels tested in this program, such an investigation for even a single steel is most time consuming, and our present study was necessarily confined to the determination of the creep behavior of these steels in the condition of heat treatment in which they are sold commercially. We have already carried out a comprehensive investigation of the influence of heat treatment on the creep strength of carbon-molybdenum steel,[10] and the fundamental principles derived therein may be applied to other alloys.

Mr. Robinson has called us to task for determining creep rates higher than those in which he is interested. The permissible total plastic deformation in the life of a turbine must be very small, and hence the creep rates of interest to turbine engineers are very low. However, high-temperature tubing is used under conditions where a larger amount of flow is permissible, and the stress for a creep rate of 1 per cent per 10,000 hr. is an accepted basic design figure employed for a considerable tonnage of steel in elevated temperature applications.

Stress-creep rate data do not necessarily lie on a straight line by any method of plotting, and extrapolation should be avoided.

In order to check the creep strength of the 5 per cent chromium-molybdenum-columbium steel C6, in the hot-rolled and tempered condition, an additional creep test was run at 1000 F. under a stress of 17,000 psi. This test showed a creep rate of 1.94×10^{-6} in. per inch per hour during the time period

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[8] Research Laboratory, U. S. Steel Corp., Kearny, N. J.

[9] Metallographist, National Works, National Tube Co., McKeesport, Pa.

[10] R. F. Miller, R. F. Campbell, R. H. Aborn, and E. C. Wright, "Influence of Heat Treatment on Creep of Carbon-Molybdenum and Chromium-Molybdenum-Silicon Steel," *Transactions*, Am. Soc. Metals, Vol. 26, p. 81 (1938).

2000 to 3000 hr. When combined with the other data, it appears that the creep rate given in Table IV for steel C6 (specimen *b*) is too high; the stress for a creep rate of 1 × 10^{-6} in. per inch per hour given for steel C6 in Table V is confirmed.

We do not wish to extrapolate the data to a creep rate of 1 per cent per 100,000 hr.

In reply to Mr. Higgins, the creep rate taken for the last thousand hours of the creep test is, in the authors' estimation, the best approximation of the creep rate which might be encountered in service under that stress and at that temperature. Depending on whether the material is growing weaker or stronger, the creep rate during the earlier period of the test may be higher or lower than the final rate.

Mr. French has interpreted our use of the term "stability" correctly. "Stable" means firmly established or permanent. We use the word in that sense to denote a structure which remains essentially unchanged during the entire test period, with no reference to any particular initial condition of the carbide particle. For instance, in carbon-molybdenum steel, the pearlitic structure is stable at 850 F., but unstable and subject to spheroidization at 1000 F. The stability of the structure varies with the composition of the steel.

We have made several attempts to determine the composition of the precipitate mentioned by Mr. Herzig. The precipitate which develops in carbon-molybdenum steel is difficult to isolate and analyze, since it is very small in amount and is pyrophoric after separation. Also, various metallographic etching reagents are of little use, since the particles are not usually resolvable under the microscope. This stable precipitate behaves in a manner very different from the iron carbide in the pearlite, which spheroidizes readily and migrates to the ferrite grain boundaries. In a previous paper,[10] we stated that this precipitate was an iron-molybdenum intermetallic compound or a complex iron-molybdenum carbide.

We appreciate Mr. Lessells' reference to the British article, and are now trying to obtain a copy of it.

Mr. Clark's comments further illustrate the need for accurate knowledge of the condition of the steel under test. Fairly good agreement was obtained when steel B3 was tested in both laboratories after the same heat treatment, considering that there was a slight difference in ferrite grain size. The structure of steel B3 shown in his Fig. 2 is known to be more creep resistant than the structure shown in Fig. 1, but it should be emphasized that this coarse grain Widmanstaetten structure is determined by air cooling from a high temperature, not by annealing from 1550 F. If this steel had been annealed from 1650 F. (the condition shown in Fig. 3) and then reannealed at 1550 F., a very different structure from that shown in Fig. 2 would have resulted.

The heat treatments used for these steels were representative of commercial practice. In regard to the modified 4 to 6 per cent chromium-molybdenum steels, we have made no investigation of the effect of variation in the titanium and columbium content.

SOME EFFECTS OF COMPOSITION AND HEAT TREATMENT ON THE HIGH-TEMPERATURE RUPTURE PROPERTIES OF FERROUS ALLOYS

By R. H. Thielemann[1]

Synopsis

This paper deals with the high-temperature rupture properties of ferrous alloys as determined by sustained load rupture testing. The general problem of the susceptibility of high-temperature materials to failure by intergranular cracking is discussed and rupture test results for 15 representative type alloys are presented. These results indicate that the susceptibility of the various alloys to failure by intergranular cracking at elevated temperatures varied with the type of the alloy as determined by its composition. It is shown from tests of fairly long duration, that the chromium-bearing ferritic type alloys are apparently the only ones that are not susceptible to failure by intergranular cracking at elevated temperature.

A system of evaluating the composition of an alloy in terms of a numerical chromium equivalent value is presented. It is pointed out that both the type of an alloy and the relative susceptibility to failure by intergranular cracking can approximately be determined by its chromium equivalent composition.

The effect of increased grain size on the high-temperature rupture strength of a low-carbon 3 per cent chromium alloy is shown. The relation of microstructure resulting from heat treatment to the high-temperature rupture properties is considered and a particular illustration of this effect is shown for a 5 per cent chromium, 1.5 per cent silicon, 0.50 per cent molybdenum alloy.

With the trend to higher temperatures and stresses, the demands placed upon materials have become increasingly severe and the known factors influencing the long-time load-carrying ability of materials are numerous indeed. Recently, the susceptibility of certain alloys to intergranular cracking at elevated temperatures has been demonstrated. It seems, with these alloys, that failure at elevated temperatures resulting from the application of load over long periods of time takes place by a process of intergranular separation along the grain boundaries. With the occurrence of this phenomenon, the normal amount of deformation preceding fracture may be greatly reduced, and there is usually no noticeable indication of the impending failure.

In high-temperature installations where stress and temperature are both known and constant, freedom from failure can, of course, be safeguarded by including ample factors of safety in design stresses which are based on the long-time creep strength of the material. But for service where operation is flexible and where the stress and temperature are known to be variable, materials should be capable of relieving stress concentrations by deformation without the occurrence of intergranular cracking. In high-temperature pressure-resisting equipment, the relief of local stress concentrations by intergranular cracking allows leaks to develop even though complete separation of the particular member has not occurred. Also, the presence of intergranular cracks, acting as stress raisers in a stressed member as at the roots of threads in high-temperature bolting, greatly reduces the safe load-carrying ability of the particular member. For these reasons it is now being recognized that for some types of service a knowledge of the long-time rupture properties of a material may be as important as a knowledge of the long-time creep strength.

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[1] Metallurgical Engineer, Research Laboratory, General Electric Co., Schenectady, N. Y.

Fortunately, some alloys do not appear to be so susceptible to intergranular cracking at elevated temperatures as do others. Results from many rupture tests on a large number of alloys indicate that the susceptibility to intergranular cracking varies with the composition of the alloy. It is found that the type of fracture incurred and the corresponding hot ductility resulting from long-time tests in the 900 to 1300 F. temperature range can be correlated with the compositions of the material. As with creep test results, such factors as heat treatment, grain size and resulting microstructure also affect the rupture test results. Certain trends and characteristics of the rupture results are believed to be indicative of the metallurgical stability and general high-temperature properties of the various alloy systems.

STATUS OF RUPTURE TESTING

As early as 1919, Jeffries[2] observed the occurrence of the two types of fractures, transcrystalline and intergranular, that result when metals are tested in tension over a wide temperature range, and postulated the idea of the equicohesive temperature. During recent years, results from high-temperature rupture tests have appeared more frequently in the literature. White, Clark, and Wilson,[3] in a recent paper, introduced the straight-line relation of stress and time to failure, which is obtained when a series of rupture test results are plotted on logarithmic coordinates. The change of slope in the rupture curve, which sometimes occurred with a change of fracture type, was shown by Thielemann and Parker[4] to be due to the accelerating influence of intergranular oxidation. Tests on annealed ingot iron in a dry hydrogen atmosphere revealed that intergranular cracking was not caused by intergranular oxidation. Although the intergranular fracture type persisted, the change of slope of the log-log plot that was found from the tests in air was not observed when the tests were made in hydrogen.

APPARATUS AND TECHNIQUE

A complete description of the rupture testing equipment employed in the Research Laboratory of the General Electric Co. appeared in a previous paper[4] and need not be repeated. At the present time, five of the twelve bar machines are in operation and tests are being conducted at 900, 1000, 1100, 1200 and 1300 F. Four of the machines are shown in Fig. 1. Various shapes and sizes of test specimens have been used, including the standard 0.505-in. tension test specimen, 0.253-in. centerless ground rods, and rectangular strips machined from sections of seamless tubing. Recently, two sizes of test specimens have

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[2] Zay Jeffries, "Effect of Temperature, Deformation, and Grain Size on the Mechanical Properties of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 60, p. 474 (1919).

[3] A. E. White, C. L. Clark and R. L. Wilson, "Fracture of Carbon Steels at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 25, p. 863 (1937).

[4] R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures After Prolonged Loading," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 135, pp. 559–582 (1939).

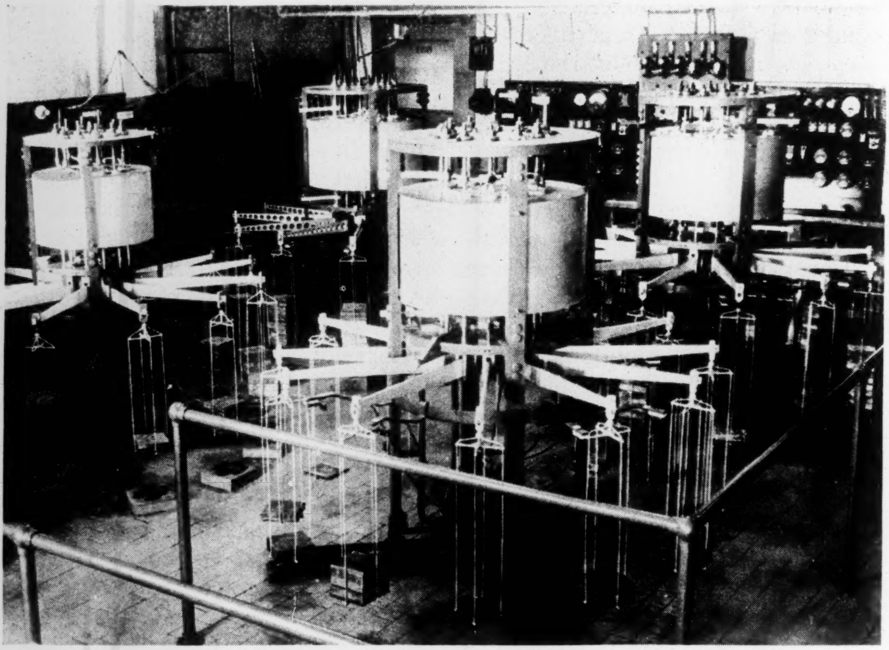
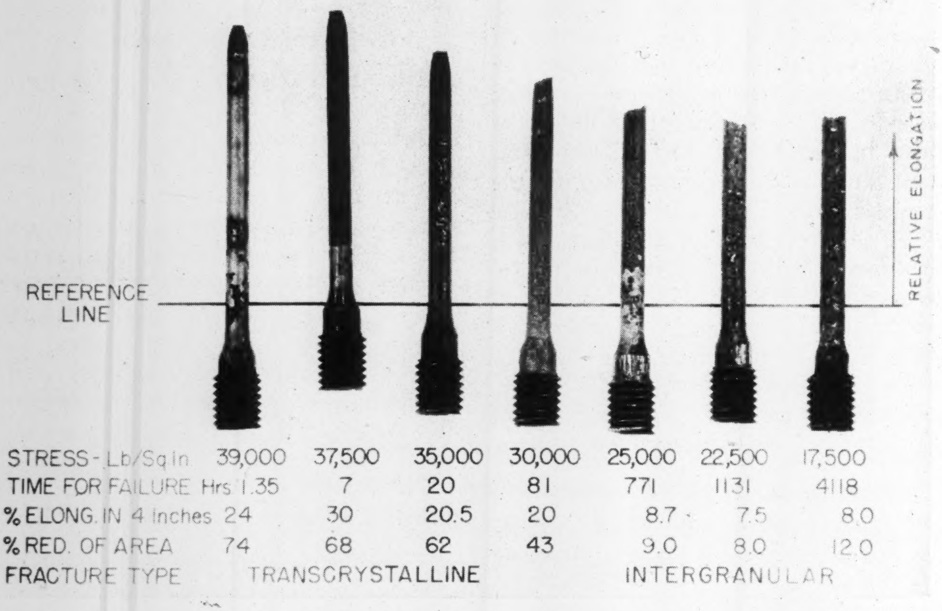


FIG. 1.—High-Temperature Sustained Load Rupture Testing Equipment.



STRESS-Lb/Sq.In.	39,000	37,500	35,000	30,000	25,000	22,500	17,500
TIME FOR FAILURE Hrs	1.35	7	20	81	771	1131	4118
% ELONG. IN 4 Inches	24	30	20.5	20	8.7	7.5	8.0
% RED. OF AREA	74	68	62	43	9.0	8.0	12.0
FRACTURE TYPE	TRANSCRYSTALLINE				INTERGRANULAR		

FIG. 2.—Appearance of Broken Test Bars from Series of Rupture Tests on Carbon-Molybdenum Steel at 1000 F.

been standardized, each employing a 4-in. gage length; one 0.253 in. in diameter and the other 0.357 in. in diameter. These threaded specimens are machined from bar stock, the larger diameter specimen being employed for tests at 1100, 1200, and 1300 F. for materials that have relatively poor resistance to oxidation. With either size of specimen, results are very consistent and where oxidation is not a factor, good agreement is found from tests of the same material using both diameters.

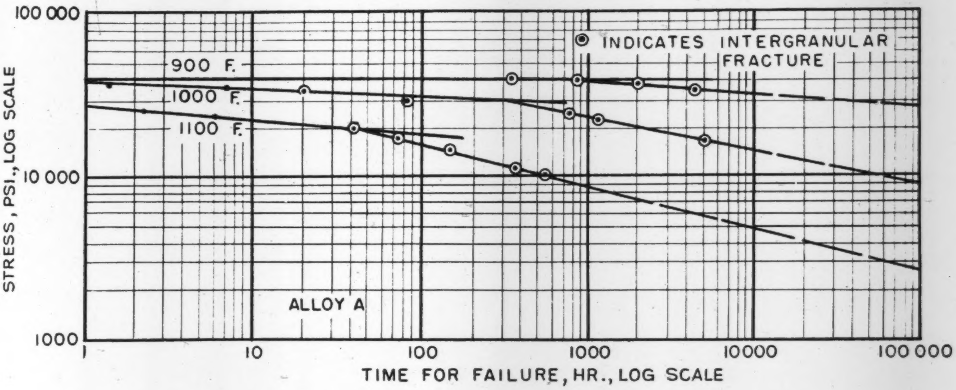


FIG. 3.—Stress-Time for Failure Curves for Carbon-Molybdenum Steel.

DISCUSSION OF RESULTS

Occurrence of the Two Fracture Types:

From external appearances, the two types of fractures encountered in the high-temperature rupture testing of ferrous alloys are characterized by the different degrees of hot ductility common to each. Ductility is here referred to as the ability of a metal to neck down and be drawn out during the process of fracturing under applied tensile loadings. Thus, transcrystalline fractures are characterized by the ductile, necked-down appearance of the broken test specimens. In contrast, intergranular fractures are marked by the lack of ductility and brittle appearance of the broken test specimens. The appearance of a series of rupture test bars of a material exhibiting both transcrystalline and intergranular fractures at 1000 F. is shown in Fig. 2. It will be noticed that with the higher values of stress, ductile transcrystalline fractures occur, and as the stresses are decreased and the corresponding fracture time is increased, the less ductile intergranular fracture is encountered.

In Fig. 3, the characteristic log-log rupture plot is shown for the tests of Fig. 2 along with the results from similar series of tests on the same material at 900 and 1100 F. It is seen that as the temperature is lowered, the slope of the log-log plot is greatly reduced. Thus, with the same stress, the time required for fracture to occur becomes increasingly longer.

Figure 4 shows the structure of this material before test. In the ductile transcrystalline fractures resulting from tests of short duration, the grains are elongated in the direction of the stress as shown in Fig. 5. Figure 6 illustrates the typical structure resulting when the fracture takes place by a process of intergranular cracking along the grain boundaries. It is evident from the equiaxed structure that elongation resulting from plastic deformation of the grains themselves has been greatly reduced.

Effect of Alloying Elements on the Resulting Fracture Type:

At an early stage of the rupture testing program it was noticed that in the 900 to 1300 F. range the susceptibility of various compositions to intergranular cracking varied appreciably with the alloy analysis. In particular, those alloys which exhibited transcrystalline fractures in tests of long duration contained appreciable amounts of chromium. In Fig. 7 are shown plotted the rupture test results for four significant alloy compositions, all of which were found to be susceptible at 1100 F., in varied degrees, to intergranular cracking. In contrast, rupture results for four other alloys tested at the same temperature, as shown in Fig. 8, indicate complete freedom from intergranular cracking in tests up to 10,000 hr. duration. Similar results for materials exhibiting both

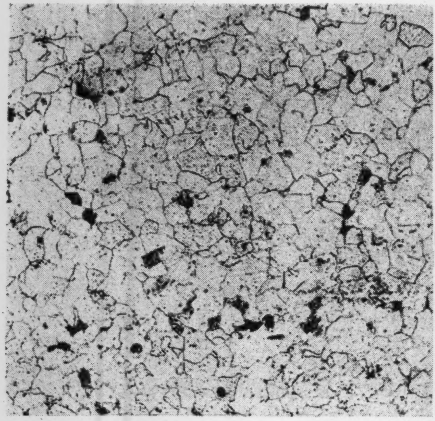


FIG. 4.—Structure of Alloy A Before Testing (× 250).

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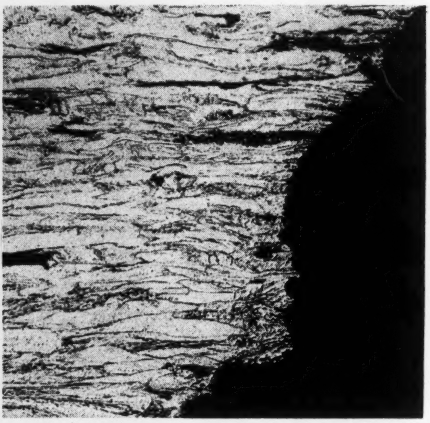

FIG. 5.—Structure of Alloy A at Fracture (× 250).

Stressed at 42,000 psi. at 900 F. Failed in 2 min. Elongation in 4 in., 25 per cent. Reduction of area, 72 per cent.

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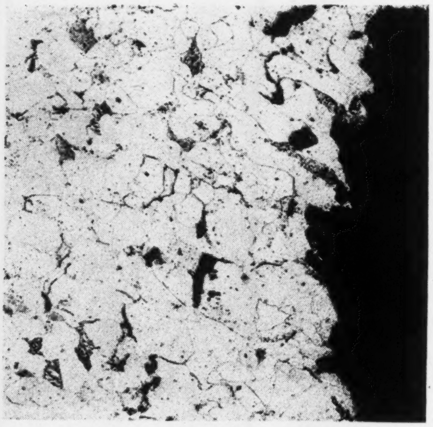

FIG. 6.—Structure of Alloy A at Fracture (× 250).

Stressed at 35,000 psi. for 4660 hr. at 900 F. Elongation in 4 in., 13.7 per cent. Reduction of area, 15 per cent.

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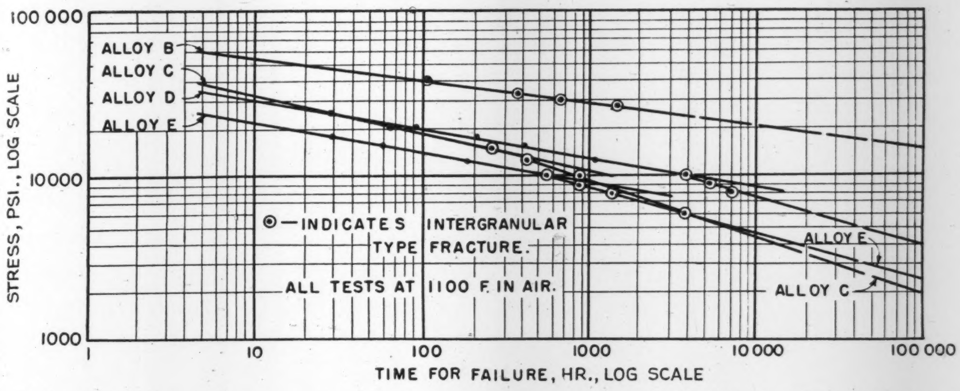

FIG. 7.—Rupture Curves for a Group of Materials at 1100 F., All of Which Are Susceptible to Intergranular Cracking.

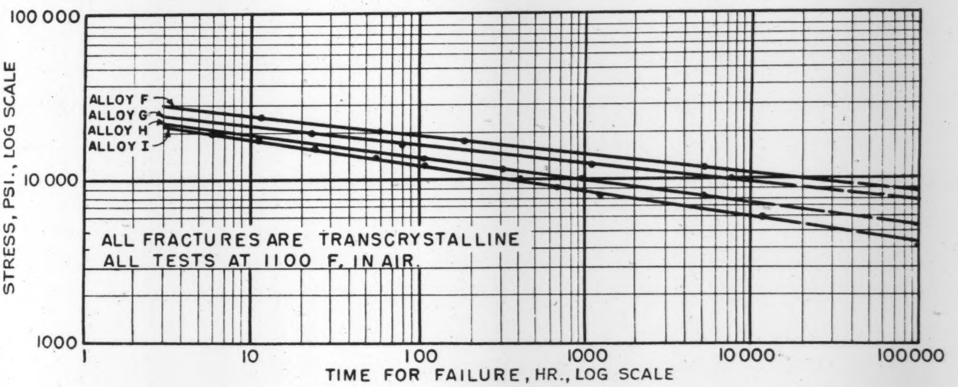

FIG. 8.—Rupture Curves for a Group of Materials, All of Which Show Continued Transcrystalline Fractures.

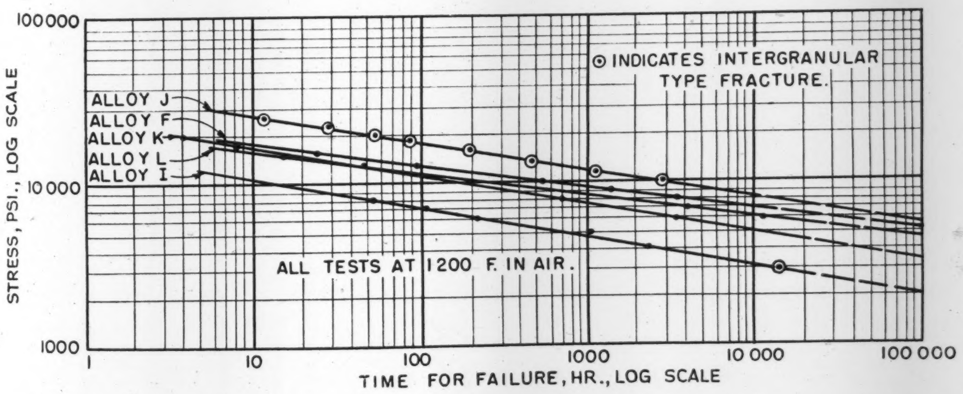

FIG. 9.—Rupture Curves for a Group of Materials Showing Both Types of Fractures at 1200 F.

types of fractures at 1200 F. are plotted in Fig. 9. For purposes of comparison, results for alloys F and I at both 1100 and 1200 F. are included in Figs. 8 and 9. These two materials are similar in composition with the exception that in alloy F the carbon content is lower and the molybdenum addition is considerably increased. At 1100 F., both materials show continued transcrystalline fractures, but at 1200 F. the long-time fractures in the case of alloy I show evidence of intergranular cracking. It is interesting to note that with the occurrence of the intergranular fracture, as shown in Fig. 9, for the 14,482 hr. fracture point, the accompanying elongation and area reduction were still ample, being 17.8 per cent in 6 in. and 37 per cent, respectively. The compositions of the various alloys included in Figs. 7, 8, and 9 are given in Table I along with the corresponding calculated chromium-equivalent values, the meaning of which will be explained later. The heat treatment and figure reference for each alloy is given in Table II.

TABLE I.—MATERIALS TESTED.

Alloy	Chemical Analysis, per cent										Chromium Equivalent
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Chromium	Nickel	Molybdenum	Tungsten	Columbium	
A	0.14	0.44	....	....	0.16	....	....	0.46	....	....	−3.8
B	0.07	0.53	0.004	0.025	0.27	19.0	9.14	....	....	0.76	−7.5
C	0.42	0.69	0.015	0.019	0.25	0.72	1.84	0.34	....	....	−20.3
D	0.12	0.29	....	....	1.23	2.97	....	0.54	....	....	+6.3
E	0.12	....	....	....	0.40	4.8	0.228	0.56	....	....	+3.8
F	0.05	0.22	0.012	0.020	1.86	5.16	....	3.42	....	....	+26.8
G	0.062	0.35	0.017	0.014	1.00	11.61	0.11	1.14	....	....	+18.0
H	0.08	0.38	0.011	0.022	0.48	13.27	0.196	....	3.22	....	+17.6
I	0.13	0.28	0.04	0.04	1.5	4.98	....	0.50	....	....	+9.1
J	0.09	0.44	0.020	0.042	0.43	19.19	9.20	0.03	....	....	−10.8
K	0.154	0.44	0.020	0.015	1.30	9.02	2.10	2.20	....	....	+11.7
L	0.05	....	....	....	1.96	5.44	....	....	3.11	....	+20.2
M	0.085	0.48	0.011	....	0.25	....	....	0.58	....	....	+0.65
N	0.065	0.47	0.010	....	0.24	....	....	1.53	....	....	+5.40
O	0.070	0.50	0.010	....	0.26	....	....	3.64	....	....	+12.80

TABLE II.

Alloy	Figure Reference	Heat Treatment
A	2, 3, 4, 5 and 6	8 hr. at 1650 F.—Furnace cooled
B	7	2 hr. at 1832 F.—Air cooled
C	7, 10 and 11	8 hr. at 1740 F.—Oil Quench—1250 F. draw
D	7, 21, 23 and 24	2 hr. at 1740 F.—Furnace cooled
D	21 and 22	Annealed at 1580 F.
E	7	Annealed at 1640 F.
F	8 and 9	Annealed at 1600 F.
G	8	8 hr. at 2280 F.—water quenched 3 hr. at 1300 F.—Furnace cooled
H	8, 12 and 13	Mill annealed
I	8, 9, 16, 17 and 18	Annealed at 1650 F.
I	25 and 26	2 hr. at 1930 F.—Air cooled. ½ hr. at 1375 F.—Air cooled
J	9, 14 and 15	2 hr. at 1832 F.—Air cooled
K	9	2 hr. at 1832 F.—Air cooled
L	9	2 hr. at 1300 F.—Air cooled
M	20	Annealed 1 hr. at 1500 F.
N	20	Annealed 1 hr. at 1500 F.
O	20	Annealed 1 hr. at 1500 F.

Micrographs corresponding to the results shown in Figs. 7, 8, and 9 are shown in Figs. 10 to 18, inclusive. Because it was found that the corresponding microstructures for the alloys of similar composition and type were almost identical, micrographs for only one composition characteristic of each of the various types are included. These figures are self explanatory, and need only to be reviewed. Figure 10 shows the acicular structure of alloy C before test and Fig. 11 shows the characteristic intergranular type of fracture resulting from tests of even short duration. It is noted also, that for the particular fracture at 1100 F., considerable spheroidization has occurred in the 888 hr. of testing time. This indicates that the acicular structure developed by heat treatment of this material is not stable under these conditions of temperature and stress. Figures 12 and 13 illustrate the characteristic before and after structures for alloys exhibiting continued transcrystalline fractures. Alloy H is of the ferritic type and from the tests at 1100 F. shows

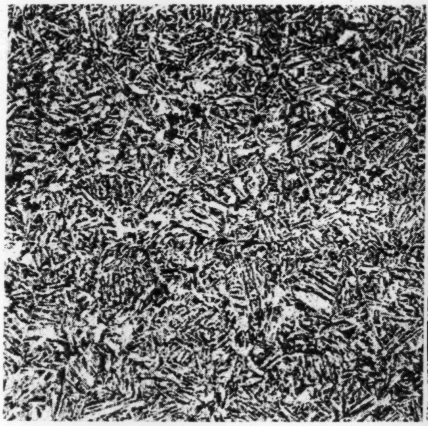

FIG. 10.—Structure of Alloy C Before Testing (× 250).

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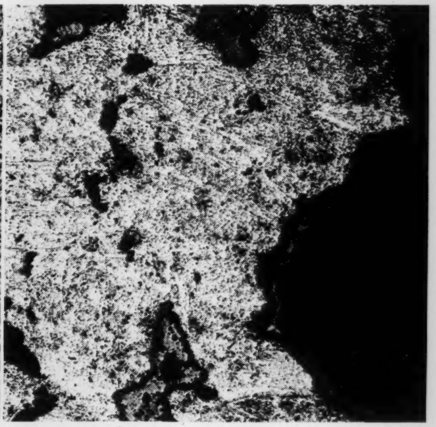

FIG. 11.—Structure of Alloy C at Fracture (× 250).

Stressed at 10,000 psi. for 888 hr. at 1100 F. Elongation in 4 in., 27 per cent. Reduction of area, 40 per cent.

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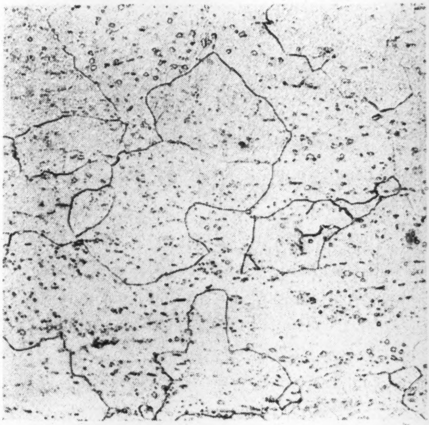

FIG. 12.—Structure of Alloy H Before Testing (× 250).

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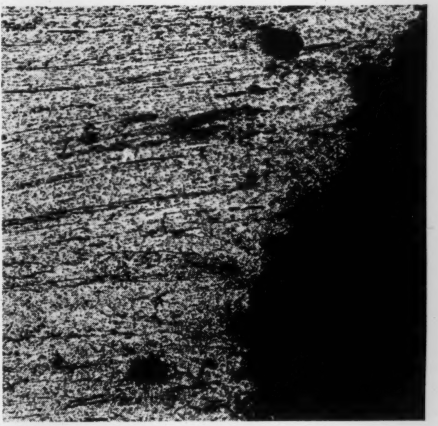

FIG. 13.—Structure of Alloy H at Fracture (× 250).

Stressed at 8000 psi. for 5240 hr. at 1100 F. Elongation in 6 in., 32 per cent. Reduction of area, 85 per cent.

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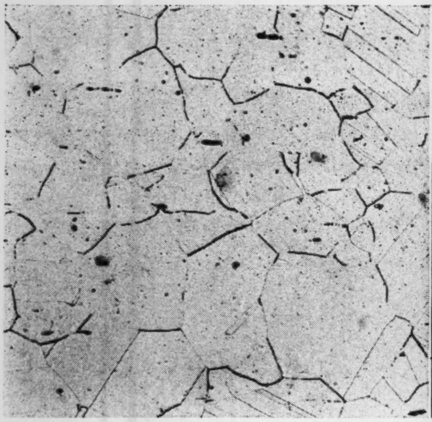

FIG. 14.—Structure of Alloy J Before Testing (× 250).

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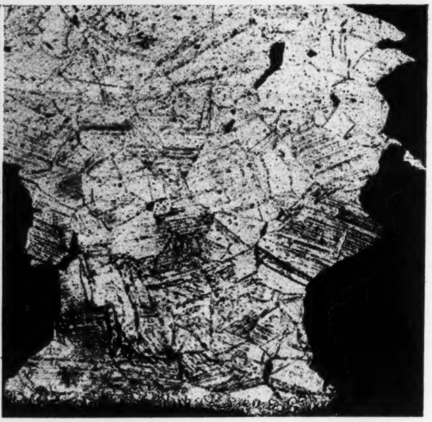

FIG. 15.—Structure of Alloy J at Fracture (× 250).

Stressed at 10,000 psi. for 2880 hr. at 1200 F. Elongation in 6 in., 27 per cent. Reduction of area, 5 5 per cent.

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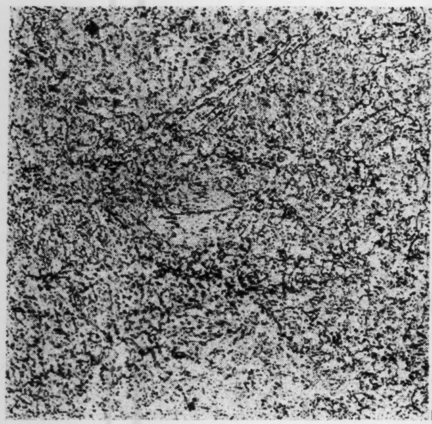

FIG. 16.—Structure of Alloy I Before Testing (× 250).

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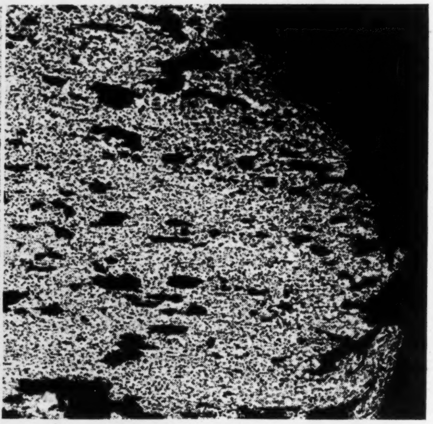

FIG. 17.—Structure of Alloy I at Fracture (× 250).

Stressed at 4000 psi. for 2346 hr. at 1200 F. Elongation in 6 in., 38.5 per cent. Reduction of area, 75 per cent.

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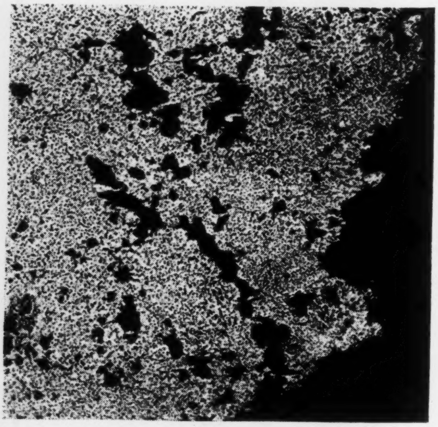

FIG. 18.—Structure of Alloy I at Fracture (× 250).

Stressed at 3500 psi. for 14,482 hr. at 1200 F. Elongation in 6 in., 17.8 per cent. Reduction of area, 37 per cent.

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no evidence of intergranular cracking whatsoever.

A typical equiaxed structure of the austenitic alloys and the characteristic intergranular fracture resulting is shown in Figs. 14 and 15, respectively, for alloy J.

Figures 16, 17, and 18 show, respectively, the original structure, the transcrystalline and the intergranular fractures for alloy I. The original structure indicates the presence of many carbides, and in this respect this alloy is different from the ferritic materials which gave no evidence of intergranular cracking under the same testing conditions.

From a review of the foregoing results, it is apparent that the materials containing appreciable amounts of only those alloying elements which tend to stabilize the alpha (body centered cubic) structure of iron, such as chromium, tungsten, molybdenum and silicon, seem to exhibit the ductile transcrystalline type of fracture. The alloys which failed by intergranular cracking contained either smaller amounts of the ferrite-forming elements, or in addition, appreciable percentages of the elements which tend to stabilize the gamma (face centered cubic) structure of iron, such as carbon, nickel, and manganese. Alloys F, G, H, and L, which are ferritic in type and practically nonhardenable by heat treatment, showed complete immunity to intergranular cracking, whereas the two austenitic alloys B and J showed intergranular fractures even in tests of only a few hours' duration. Going from the ferritic to the austenitic type in the order named, the four heat-treatable alloys K, I, D, and E also exhibited increased susceptibility to intergranular cracking. Thus it seems that for various materials the type of fracture at elevated temperatures resulting from a sustained tensile load can be correlated with the type of the particular alloy in question as determined by its alloy composition.

Fracture Type versus Chromium Equivalent Composition:

In order to establish a numerical system of values by which the relative susceptibilities of the various alloys to intergranular cracking at elevated temperatures can be expressed, it is necessary to review the effects of the various alloying elements on the structure of iron as shown by the numerous constitution diagrams. It was shown that only the ferritic type of alloys exhibited continued transcrystalline fractures in the tests of reasonably long duration. By virtue of their composition, these so-called alpha-delta alloys are nonhardenable and lie outside of the gamma loop of their respective constitution diagrams. In the iron-chromium system it is quite well established that an addition of approximately 13 per cent of chromium[5] will completely eliminate the gamma form of iron and produce an alloy of the same nonhardenable alpha-delta type. Similarly, an addition of 6 per cent of tungsten, 3 per cent of molybdenum or 2.5 per cent of silicon by weight will render the same result. It follows that tungsten is 13/6 or 2.1 times more effective than chromium in narrowing the temperature range of the stable gamma phase in iron and on the basis of equal weight the chromium equivalent for tungsten is 2.1. Similarly, chromium equivalent values for molybdenum and silicon are 4.2 and 5.2, respectively. Additions of those elements which tend to widen the temperature range of the gamma structure of iron have the effect of shifting the extremity of the closed gamma loop to higher percentages of the ferrite stabilizing elements. Investigations of a large number of alloy compositions have indicated the relative amounts of chromium, tungsten, molybdenum and silicon necessary to offset increased additions of carbon, nickel, and manganese. From these results, approximate chromium equivalent values for the austenite-forming elements have been determined to be as follows: carbon, −40; nickel, −3; and manganese −2. The negative sign indicates the opposite effect of these elements with respect to the action of chromium.

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[5] Metals Handbook, 1939 Edition, p. 374, Am. Soc. Metals.

It is convenient to express the relative effects of the various alloying elements in terms of the effectiveness of chromium because of its common presence in practically all alloys intended for service at elevated temperatures and because it is the least effective of the common alloying elements in eliminating the gamma phase in iron. In Table III chromium equivalent values for several of the alloying elements are tabulated. The two elements, tantalum[6] and columbium[7] apparently do not form closed gamma loops but up to their limits of solubility in alpha iron have the effect of narrowing the temperature range of the gamma field, as indicated by their corresponding chromium equivalents. Cobalt, except when present in large amounts, shows only a slight tendency to stabilize the gamma structure in iron.

TABLE III.—CHROMIUM EQUIVALENT VALUES FOR ALLOYING ELEMENTS IN IRON.

Element	Lattice Type	Effect on Temperature Range of Gamma Phase[c]	Percentage by Weight to Eliminate Gamma Phase	Chromium Equivalent
Carbon	Amorphous	Widens		−40
Nickel	Face centered cubic	Widens		−3
Manganese	Cubic	Widens		−2
Copper	Face centered cubic	Widens		−1
Cobalt	Hexagonal close packed	Widens (has weak effect)		
Chromium	Body centered cubic	Narrows	13	+1
Tungsten		Narrows	6	+2.1
Tantalum[a]		Narrows up to 1%		+2.8
Molybdenum		Narrows	3	+4.2
Columbium[b]		Narrows up to 1.8%		+4.5
Silicon	Cubic	Narrows	2.5	+5.2
Titanium	Body centered cubic	Narrows	1.8	+7.2
Vanadium		Narrows	1.2	+11.0
Aluminum	Face centered cubic	Narrows	1.1	+12.0

[a] Maximum solubility in alpha iron is about 1 per cent at 974 C. (1785 F.), see Metals Handbook, 1939 Edition, p. 359, Am. Soc. Metals.

[b] Maximum solubility in alpha iron is about 1.8 per cent at 975 C. (1785 F.), see R. Genders, "Iron Niobium Alloys," *Engineering*, Vol. 148, November 3, 1939, pp. 514 and 515.

[c] See E. C. Bain, "Alloying Elements in Steel," Am. Soc. Metals, pp. 9, 10, 11 (1939).

In a large number of various alloys including the ones reported here the separate effects of the alloying elements in narrowing or widening the limits of the gamma field in iron are found to be cumulative. As an example, additions of either molybdenum or silicon or of both to a 5 per cent chromium iron alloy apparently have the same effect in rendering a closed gamma loop as would an equivalent addition of chromium. Thus the chromium equivalent value for a given composition can be determined by adding together the individual chromium equivalent percentages of the various alloying elements present. It follows that alloys having the same chromium equivalent composition will be similar with respect to their position in the fields outlined by the gamma loop.

The relation of the chromium equivalent composition to the type of alloy resulting is shown diagrammatically in Fig. 19. With respect to the type of fracture encountered from long-time high-temperature rupture tests, alloys having a chromium equivalent composition greater than (+13) show continued transcrystalline fractures in the 900 F. to 1300 F. temperature range. Alloys having chromium equivalent compositions of about +9 to +13 lie in the two-phase alpha + gamma field, and, in the annealed condition, show intergranular type fractures only from tests of long duration. With these borderline alloys the minimum values of elongation accompanying the intergranular fractures still seem to be appreciable. With decreasing chromium equivalent values, the resulting alloys become increasingly susceptible to intergranular cracking. The elongation accompanying long-time fractures varies with the heat treatment and with the composition. With the completely austenitic type of alloy, brittle intergranular fractures are found even from tests of short duration.

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[6] Metals Handbook, 1939 Edition, p. 359, Am. Soc. Metals.

[7] R. Genders, "Iron Niobium Alloys," *Engineering* Vol. 148, November 3, 1939, pp. 514 and 515.

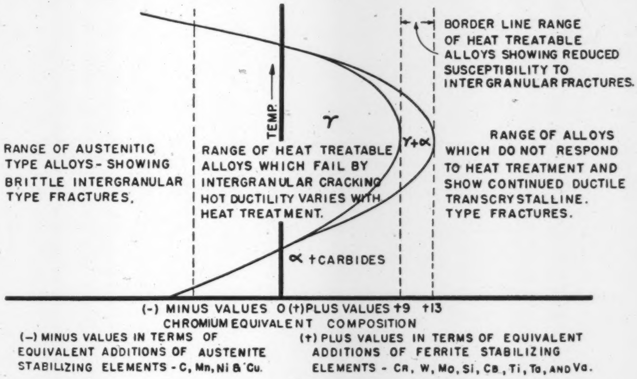
BORDER LINE RANGE OF HEAT TREATABLE ALLOYS SHOWING REDUCED SUSCEPTIBILITY TO INTER GRANULAR FRACTURES.
TEMP.
γ
$\gamma+\alpha$
RANGE OF AUSTENITIC TYPE ALLOYS - SHOWING BRITTLE INTERGRANULAR TYPE FRACTURES.
RANGE OF HEAT TREATABLE ALLOYS WHICH FAIL BY INTERGRANULAR CRACKING HOT DUCTILITY VARIES WITH HEAT TREATMENT.
RANGE OF ALLOYS WHICH DO NOT RESPOND TO HEAT TREATMENT AND SHOW CONTINUED DUCTILE TRANSCRYSTALLINE. TYPE FRACTURES.
α +CARBIDES
(-) MINUS VALUES 0 (+) PLUS VALUES +9 +13
CHROMIUM EQUIVALENT COMPOSITION
(-) MINUS VALUES IN TERMS OF EQUIVALENT ADDITIONS OF AUSTENITE STABILIZING ELEMENTS - C, Mn, Ni & Cu.
(+) PLUS VALUES IN TERMS OF EQUIVALENT ADDITIONS OF FERRITE STABILIZING ELEMENTS - CR, W, MO, SI, CB, TI, TA, AND VA.

FIG. 19.—Diagrammatic Sketch Showing Relation of Chromium Equivalent Composition of Alloys to Fracture Type Incurred from Long-Time Sustained Load Rupture Tests in 900 F. to 1300 F. Range.

In applying the chromium equivalent system to alloys of various compositions, it is realized that irregularities are to be found in certain of the alloy systems. In the first place, constitution diagrams for some of the various alloy systems are not well established, and, when more information is available, the chromium equivalent values for some of the alloying elements may have to be revised. Secondly, there are indications that the chromium equivalent values for carbon and the other austenite stabilizing elements may vary somewhat with the different alloy systems. Also, due to the appearance of new phases, it is apparent that the chromium equivalent values will apply only when the maximum solubility limits of the alpha stabilizing elements in iron are not exceeded. However, the present chromium equivalent values have been applied to a large number of alloys with a high degree of success. It has been possible to design alloys for certain high-temperature applications which would be less susceptible to brittle intergranular cracking without sacrificing other desirable properties. It is found, for instance, that with the alpha-delta type of alloy, having chromium equivalent compositions in excess of +13, large ferritic grains resulting in extreme room temperature brittleness are developed from overheating, as in welding operations. This brittleness could not be relieved by heat treatment; so in order to maintain a heat-treatable alloy which could be fabricated by welding and would still possess a high degree of freedom from intergranular cracking, the chromium equivalent composition was limited to a value of +11.

Restrictions in Composition:

All of the alloys thus far mentioned whose chromium equivalent compositions were in excess of +9 contained at least 5 per cent of chromium. The most susceptible of these alloys showed intergranular fractures only in tests of long duration. It appears that an addition of 4 or 5 per cent of chromium is necessary in any ferrous alloy, regardless of its chromium equivalent composition, if complete freedom from intergranular cracking is to be insured. Rupture test results in Fig. 20 show the effect of molybdenum additions on the rupture properties at 1100 F. of three carbon-molybdenum alloys M, N, and O whose chromium equivalent compositions are +0.65, +5.4, and +12.8, respectively. The two alloys M and N, with molybdenum contents of 0.58 and 1.53 per cent, showed intergranular fractures even for tests of short duration. The 3.64 per cent molybdenum alloy, O, was much less susceptible to intergranular cracking. However, in comparison with chromium-

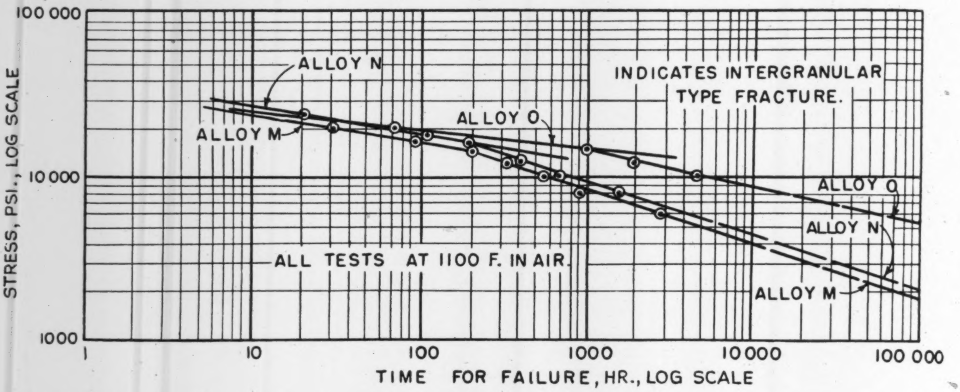

FIG. 20.—Rupture Curves Showing Effect of Increasing Additions of Molybdenum on Rupture Properties of Carbon-Molybdenum Steel at 1100 F.

TABLE IV.—CORRELATION OF FRACTURE TYPE WITH CHROMIUM EQUIVALENT OF ALLOYS TESTED.

Alloy	Chromium Equivalent	Results from Longest Rupture Test					
		Temperature, deg. Fahr.	Stress, psi.	Time for Fracture, hr.	Elongation in 6 in., per cent	Reduction of Area, per cent	Type of Fracture
F..........	+26.8	1100	12 500	5 225	17.8	78.0	transcrystalline
F..........	+26.8	1200	8 000	3 455	22.0	73.0	
L..........	+20.2	1200	6 000	11 676	32.5 in 4 in.	78.0	
G..........	+18.0	1100	10 000	7 497	15.6	78.5	
H..........	+17.6	1100	8 000	5 240	32.0	85.0	
O..........	+12.8	1100	10 000	4 506	14.6	37.0	intergranular
K..........	+11.7	1200	6 000	3 598	23.0	83.5	transcrystalline
I..........	+9.1	1100	6 000	11 575	30.0	81.0	
I..........	+9.1	1200	3 500	14 482	17.8	37.0	intergranular
D..........	+6.3	1100	8 000	6 848	14.6	50.0	
N..........	+5.4	1100	8 000	1 589	4.1	4.0	
E..........	+3.8	1100	6 000	3 882	10.0	37.0	
M..........	+0.65	1100	6 000	2 712	7.0	18.0	
A..........	−3.8	900	35 000	4 660	13.7 in 4 in.	15.0	
A..........	−3.8	1000	17 500	4 118	8.0 in 4 in.	12.0	
A..........	−3.8	1100	10 000	557	18.7 in 4 in.	49.0	
B..........	−7.5	1100	27 500	1 607	3.5	4.0	
J..........	−10.8	1200	10 000	2 880	2.0	5.5	
C..........	−20.3	1100	10 000	888	27.0 in 4 in.	40.0	

bearing alloys of even lower chromium equivalent compositions, such as alloy I in Fig. 8, it is more susceptible to intergranular cracking.

In Table IV the alloys listed in Table I are retabulated in the order of decreasing chromium equivalent compositions. For each alloy the fracture time, elongation, reduction of area, and corresponding fracture type are given for the tests of longest duration. From this correlation it seems evident that the long-time fracture types for all alloys having chromium equivalent compositions below +9 will be of the intergranular type. In the higher chromium equivalent compositions the fracture types are decidedly transcrystalline, the one exception being the straight 3.6 per cent molybdenum alloy O which contained no chromium.

Effect of Grain Size on Rupture Strength:

In general, the effect of increasing the grain size of fine-grained alloys by heat treatment is to increase the corresponding rupture strength. Weaver[8] has shown a similar effect of increased grain size on the resulting creep strength. Figure 21 shows the effect of increased grain size on the rupture properties of a 3 per cent chromium alloy (alloy D) at 1100 F. Aside from the increase in rupture strength, it seems that the larger grain size has the effect of decreasing the susceptibility of the alloy to intergranular cracking at this temperature. With the fine-grained material intergranular fractures were found for all tests over 300 hr. duration whereas with the larger grain size the change of fracture type was noted only for the tests of more than 3000 hr. duration. Of greatest significance is the fact that, by increasing the grain size, the indicated 100,000-hr. rupture strength has been almost doubled. The original fine-grained structure and the corresponding large-grained structure resulting from heat treatment are shown in Figs. 22 and 23, respectively.

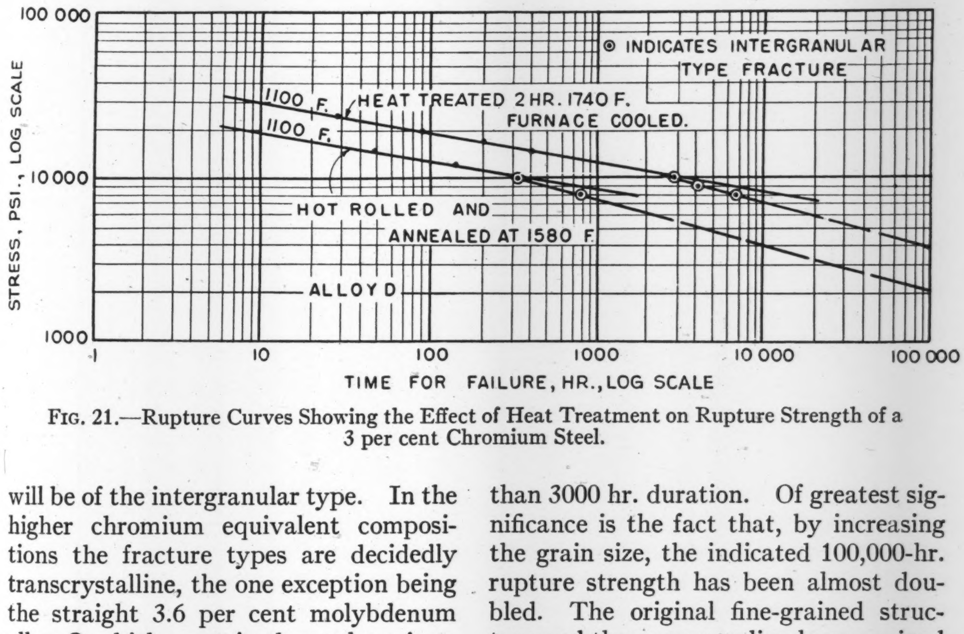

FIG. 21.—Rupture Curves Showing the Effect of Heat Treatment on Rupture Strength of a 3 per cent Chromium Steel.

The structure at the fracture for the test of longest duration, as shown in Fig. 24, indicates elongated grains with only partial grain boundary separation. Since the chromium equivalent composition for this alloy is +6.5, more completely intergranular fractures would result from tests of longer duration.

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[8] S. H. Weaver, "Actual Grain Size Related to Creep Strength of Steels at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 176 (1938).

Effect of Structure on Fracture Type:

Previously it was mentioned that the borderline alloys having chromium equivalent compositions in the +9 to +13 range showed considerable freedom from intergranular cracking when tested in the annealed condition. Because this group of alloys is responsive to heat treatment, it is logical to expect that the structural changes resulting might have some effect on the rupture properties. Figure 25 shows the acicular structure developed in alloy I obtained by air cooling through the critical range. The $\frac{1}{2}$-in. diameter bar stock was heated first for 2 hr. at 1930 F. and air cooled and then reheated for $\frac{1}{2}$ hr. to 1375 F. and air cooled. The Rockwell hardness, B scale, of the original structure shown in Fig. 19 was 88. After quenching in air, the

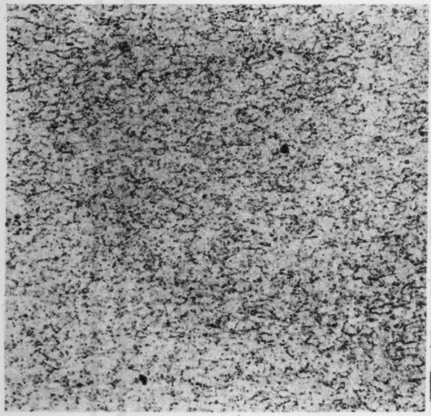

FIG. 22.—Original Fine-Grained Structure of Alloy D (× 250).
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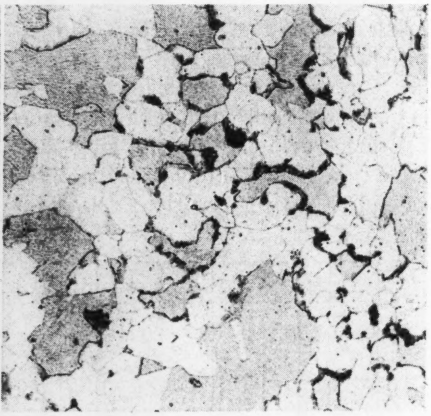

FIG. 23.—Heat-Treated Structure of Alloy D Before Testing (× 250).
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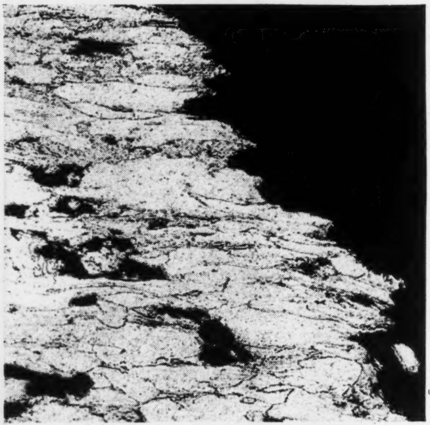

FIG. 24.—Structure at Fracture of Heat-Treated Alloy D (× 250).
Stressed at 8000 psi. for 6848 hr. at 1100 F. Elongation in 6 in., 14.6 per cent. Reduction of area, 5 per cent. Reduced to ½ size in reproduction.

hardness increased to 108, indicating that the material is air hardening. The $\frac{1}{2}$-hr. draw at 1375 F. reduced the hardness to 89 so that the hardness of the structure shown in Fig. 25 is almost the same as that of the original structure. Figure 26 shows the intergranular fracture which is found from rupture tests on this material in the heat-treated condition. This specimen was stressed at 6000 psi. for 1150 hr. at 1200 F. before fracture occurred. The elongation was 16.8 per cent in 6 in. with 29 per cent reduction of area. In comparison, a bar of the original material stressed at 5000 psi. at the same temperature failed with a transcrystalline fracture after 1010 hr. The accompanying elongation and reduction of area were 30 per cent in 6 in. and 83.5 per cent, respectively. Apparently, with these borderline alloys, the resistance of the grain boundaries to separation at elevated temperatures is not much different from the resistance of the grains to deformation by shear along the preferential planes of slip of the crystal lattices. Thus, with this particular alloy, the effect of the acicular structure as developed by heat treatment was to strengthen the grains to the extent that they were more resistant to continued deformation by shear than were the grain boundaries to separation by intergranular cracking. By the same reasoning it seems that the strengthening effect of the various hard carbides which result from heat treatment with the materials of lower chromium equivalent compositions could account for the increased susceptibility of these materials to intergranular cracking.

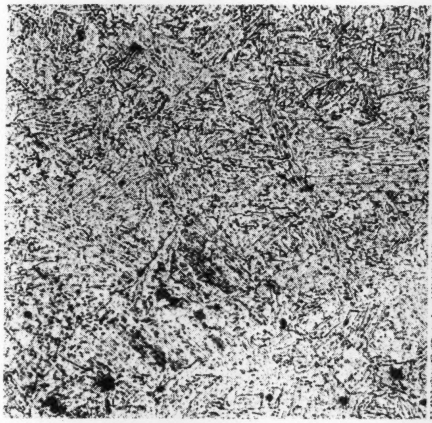

FIG. 25.—Heat-Treated Structure of Alloy I Before Testing (× 250).
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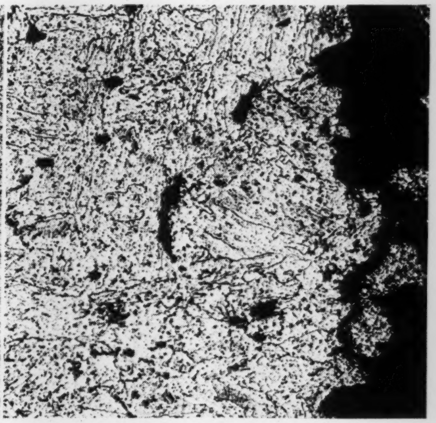

FIG. 26.—Structure at Fracture of Heat-Treated Alloy I (× 250).
Stressed at 6000 psi. for 1150 hr. at 1200 F. Elongation in 6 in., 16.8 per cent. Reduction of area, 29 per cent.
Reduced to ½ size in reproduction.

CONCLUSIONS

From sustained load rupture tests on various ferrous alloys at elevated temperatures both transcrystalline and intergranular fractures are encountered. Transcrystalline fractures are characterized by the ductile, necked down appearance of the broken test specimens. In contrast, intergranular fractures are marked by their lack of ductility and brittle appearance.

As evidenced by the resulting microstructure, plastic deformation resulting in ductile transcrystalline fractures takes place by a process of slip movement along certain crystallographic planes. The relief of strain hardening, by continual recrystallization with time, allows this type of deformation to progress. If the resistance of the polycrystalline grains to continual deformation by slip is greater than the resistance of the grain boundaries to separation, intergranular fractures will result. Higher temperatures and lower strain rates favor the occurrence of this type of fracture.

The susceptibility of the various alloys to failure by intergranular cracking varies with the type of the alloy as determined by its composition. Only the chromium-bearing ferritic alloys did not show evidence of intergranular cracking in tests of long duration. With the nickel-chromium austenitic alloys, brittle intergranular fractures are encountered even in tests of only a few hours duration. The rupture properties of those alloys which will respond to heat treatment can be varied by altering the structures within the grains. A system by which the chromium equivalent composition of an alloy can be calculated is found to be useful in determining its relative susceptibility to failure by intergranular cracking at elevated temperatures.

As with the creep strength, the effect of increasing the grain size of an originally fine-grained alloy is to increase its rupture strength.

Acknowledgments:

Several persons have contributed to the information presented in this paper. The chromium equivalent system was first proposed by G. R. Brophy in 1934 as a means of classifying hardenable and nonhardenable alloys. To A. J. Nerad is credited the idea of correlating the high-temperature fracture type with the chromium equivalent composition of the alloy. Sincere appreciation is given to E. L. Thearle for his generous assistance in directing this work and to L. L. Wyman for the preparation of the photomicrographs.

DISCUSSION

MR. T. McLEAN JASPER[1] (*presented in written form*).—The writer believes that the general method of testing presented in the paper can be easily applied by the engineer and for this reason will eventually attain premier position for tests at elevated temperatures.

The very large amount of activity which has centered around testing steels at high temperatures has allowed a very careful scrutiny of methods and results to be made. It might be pointed out that in general the testing engineer is prone critically to investigate the material tested and not associate the results in some measure with the environment of testing or with the measuring equipment or relative size of specimens, etc. In general, when steel is used at elevated temperatures its cross-section is of a different order from that of a small test specimen. The oxidation of a small specimen may affect the results to a degree which will mislead the engineer in designing equipment in which oxidation plays but a relatively minor part in service.

The writer is primarily interested in low-alloy steels in the design and building of equipment for high-temperature service in which some form of additional corrosion protection is frequently used. My purpose in drawing attention to the above is to help keep the attitude toward methods of testing subservient to the requirements of the structure as used in service.

MESSRS. C. L. CLARK,[2] A. E. WHITE,[3] AND MARTIN FLEISCHMANN[4] (*presented in written form*).—The author is to be congratulated on this contribution to the behavior of metals at elevated temperatures and especially to the influence of certain factors on the prolonged rupture strength of steels at temperatures of 900 to 1200 F., inclusive.

In most respects the results and conclusions drawn from our own rupture tests are in agreement with those of the author, but certain exceptions do exist which it is believed should be presented in order to show the inadvisability of drawing too broad conclusions at this time.

It is agreed that the intergranular type failures always produce a blunt type fracture, but we have not found this type fracture to be always associated with brittleness, as indicated by the total elongation up to fracture. For example, our work on annealed 0.50 per cent molybdenum steel shows that for a fracture time of 4000 hr., the elongation at 950 and 1000 F. is very low, being of the order of 5 per cent, while at 1100 and 1200 F., it is 15 per cent. Even this latter value is relatively low but it is apparent that for the same type fracture the elongation at the higher temperatures is three times that at the lower temperatures. Furthermore, the value of 15 per cent corresponds to certain of those given in Table IV as resulting from the transcrystalline type of failure.

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[4] Metallurgical Engineer, The Timken Roller Bearing Co., Canton, Ohio.

Likewise, in prolonged tests conducted on a steel of the 1.25 per cent chromium, 0.50 per cent molybdenum, 1.35 per cent silicon type, intergranular type failures and blunt fractures were obtained at both 900 and 1100 F. Yet, for a fracture time of 11,000 hr. at 900 F. the elongation was 17 per cent, and after 14,000 hr. at 1100 F., it was 42.5 per cent.

Many other examples could be presented, but those given are believed sufficient to show that intergranular failures and blunt fractures do not necessarily result in a low ductility, as measured by the elongation, at temperatures of 900 to 1100 F. It should be further recognized that at certain temperatures in excess of those considered in this paper the fracture will change to the intergranular type even in the steels which exhibited transcrystalline failure at 1200 F. Again, however, this does not necessarily imply that these steels would become brittle, for in most cases we have obtained very satisfactory ductility characteristics at the more elevated temperatures.

The statement that the presence of 4 to 5 per cent of chromium is necessary to insure against intergranular cracking should be modified so as to specify temperature. For example, tests at 1000 F. up to 6000 hr. on a 1.25 per cent chromium, 0.50 per cent molybdenum, 0.75 per cent silicon steel showed transcrystalline fractures to exist, with no tendency for intergranular cracking and with an elongation of 28.5 per cent. At 1200 F. this same steel does show an intercrystalline fracture but after 4000 hr. the elongation is high, being 46 per cent.

Considerable work has been done in our laboratories on the combined influence of ferrite-forming elements on the gamma loop and it is expected that these findings will soon be published. The indications are that in the presence of carbon these elements do not exert a strictly additive effect on the gamma loop. Likewise it is believed that the region of alpha plus gamma in Fig. 19 should be greatly broadened, for we have found that with 0.15 per cent carbon present, this mixed phase occupies an area several times as wide as that of the homogeneous gamma region. Using the author's method of computing equivalent chromium content, we have found that in one series of steels the inner gamma loop is closed at an equivalent chromium content of approximately 18.0 per cent and the two-phase area is not closed even with an equivalent chromium of 30.0 per cent.

MR. ARTHUR MCCUTCHAN[5] (*presented in written form*).—The author has evolved an ingenious method of correlating the high-temperature fracture type with the chromium equivalent composition of the material. This method of correlation, however, can be misleading. The author's conclusions would appear to disqualify a chromium-silicon-molybdenum material which has shown good promise as a pipe and tube material at temperatures of 1000 to 1100 F. This material has the following chemical composition:

Carbon, per cent	0.15 max.
Manganese, per cent	0.30–0.60
Chromium, per cent	1.00–1.50
Molybdenum, per cent	0.45–0.65
Phosphorus, per cent	0.03 max.
Sulfur, per cent	0.03 max.

From the chromium-equivalent correlation made by the author this material should show poor hot ductility, that is, failure under prolonged stress-rupture tests should show brittle failure at 900 to 1000 F. Actually, good elongation and reduction of area values are found for this material under this type of loading.

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[5] Engineer, Engineering Division, The Detroit Edison Co., Detroit, Mich.

A service test[6] on a thin-walled tube of a higher silicon version of this alloy at 1100 F. in an experimental steam line showed that a diametral extension of 7 per cent could occur without any evidence of brittle failure. Steam and air corrosion plus thinning of the wall from this extension caused an increase in working stress of over 20 per cent. Obviously this material gives ample warning of approaching failure.

Since this material is readily weldable, possesses good resistance to steam attack, and even higher creep strength than 4 to 6 per cent chromium, 0.50 per cent molybdenum steel, it would be unfortunate if it were passed over because it failed to fit the chromium-equivalent theory deduced from the series of alloys tested by the author.

One point should be amplified by the author. An impression of intergranular cracking might be gained from his discussion to the effect that a material such as carbon-molybdenum after prolonged service at 900 to 1000 F. would be found to be brittle or have low ductility if subsequently tested at room temperature. Question whether this conclusion is justified might be suggested from the following room temperature test results[7] on creep specimens which were subjected to stress at the temperatures and for the durations indicated below:

Temperature, deg. Fahr.	Stress, psi.	Duration, hr.	Tensile Strength, psi.	Yield Stress, 0.2 per cent Offset, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent
85			62 150	40 000	37.5	66.5
900	13 000	1055	61 700	39 000	38.5	65.6
900	17 500	1055	62 000	35 000	37.5	67.9
1000	8 000	1115	61 400	38 500	37.0	74.1
1000	12 000	1105	60 750	40 000	43.5	69.5

Izod tests on small V-notch specimens likewise showed an insignificant change in the physical properties of the carbon-molybdenum tension creep specimens.

While the above results were obtained on material held for only a little over 1000 hr. at temperature, they tend to show that carbon-molybdenum steel does not become brittle in the sense that cast iron is called brittle. Specifically, the writer's question might be phrased as follows: Would a tension or impact test on a specimen cut from a carbon-molybdenum pipe after 10 yr. of service, under stress and temperature conditions such that a diametral extension of three-fourths of 1 per cent had occurred, be expected to reveal a condition of brittleness or approaching failure? This distinction between "intercrystalline cracking" associated with slowly stretched metals under high-temperature conditions and "embrittlement" such as occurs in some bolting materials was discussed at some length by Rosenhain over 8 yr. ago.[8]

In conclusion, the author should be commended for his endeavor to correlate type of fracture with chemical composition. The possibility of nonductile behavior under high-temperature loadings is extremely important and the writer has a high regard for the value of this contribution to the meager knowledge of this subject.

MR. R. F. MILLER.[9]—The results of some of our creep and rupture tests on carbon, 0.5 per cent molybdenum steel may be of interest in comparison with those obtained by Mr. Thielemann.

A specimen of this steel was normalized from 1790 F. and tested to rupture at 1020 F. under a stress of 27,000 psi. Intergranular fracture occurred after 420 hr. with an elongation of 12.2 per cent.

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[6] R. M. Van Duzer, Jr., and Arthur McCutchan, "High Temperature-Steam Experience at Detroit," *Transactions*, Am. Soc. Mechanical Engrs., July, 1939, pp. 383–401.

[7] Timken Digest of Steels for High-Temperature Service, Fourth Edition, 1939, p. 25.

[8] Walter Rosenhain, "Quick Determinations of Limiting Creep Stress," *Metal Progress*, February, 1932, p. 66.

[9] Research Laboratory, U. S. Steel Corp., Kearny, N. J.

These results were almost identical with those obtained by Jenkins and his coworkers at the National Physical Laboratory in England and agree quite well with those shown in Fig. 3 of Mr. Thielemann's paper.

Another specimen of the same steel was normalized from 1650 F. and tempered for 5 hr. at 1300 F. This specimen was tested to rupture at 1000 F. under a stress of 27,000 psi. Intergranular fracture occurred after 1155 hr. with 41.8 per cent elongation. Microscopic examination disclosed that there was considerably more distortion and elongation of the grains in this specimen than in the one described above. The greater ductility of this specimen is thought to be due to tempering at 1300 F. These different results on the same steel under the same stress at approximately the same temperature tend to bear out Mr. Thielemann's conclusions regarding the effect of structure on fracture type.

Both of these specimens had quite high loads applied at the start of the test and both showed intergranular fracture. However, in the three specimens now to be described, the loads were applied in two steps and transcrystalline (ductile) failures were observed in all three. In the first of these tests, a specimen of carbon, 0.5 per cent molybdenum steel was normalized from 1650 F. and tempered for 5 hr. at 1300 F. It was subjected to a load of 15,000 psi. for 3000 hr. at 1000 F., producing 3.85 per cent elongation. The load was then raised to 27,000 psi., and transcrystalline fracture occurred after 12 hr. with 30 per cent elongation. The second specimen of this steel had the same heat treatment and was subjected to a load of 3500 psi. at 1100 F. for 14,000 hr., producing 2.2 per cent elongation. The load was then raised to 7500 psi., and transcrystalline fracture occurred in 1000 hr. with 74.0 per cent elongation. In the third test, a specimen of carbon, 0.5 per cent molybdenum steel was normalized from 1650 F. and tempered for 168 hr. at 1300 F. It was subjected to a stress of 5000 psi. for 3500 hr. at 1100 F., producing an elongation of 4.2 per cent. The stress was then raised to 7500 psi., producing transcrystalline failure after 700 hr. with 103 per cent elongation.

The results of these three latter tests make it apparent that under working conditions of over-heating or over-stressing, carbon-molybdenum steel may have greater ductility than the high-stress creep-to-rupture tests would lead us to expect. As Mr. Thielemann has pointed out, the relative ductility and type of fracture of a given steel at a given temperature depend on its heat treatment and resulting microstructure. It should be borne in mind that exposure to service temperatures of 1000 or 1100 F. for even a few thousand hours is in itself a form of heat treatment, and may alter the structure and properties of the material. Creep-to-rupture tests may therefore be misleading, unless they are of sufficient duration to produce structural changes similar to those found in service.

MR. A. NADAI.[10]—I would like to ask whether a vacuum creep test has been made and whether the presence of air influences creep. If it does I would ask a second question, as to whether the size of the surface or, in other words the size of the test specimens, also influences the results obtained in creep tests.

MR. R. H. THIELEMANN[11] (*Author's closure*).—The comments and constructive criticisms from those who have discussed this paper are deeply appreciated. Mr. Jasper's suggestion, that the testing engineer is prone critically to investigate the material tested and not associate the results in some manner with the size of the test specimens and conditions of testing, is in many cases true. We have found, with materials which are subject to oxidation and scaling at the testing temperature, that the time required for failure with a given stress is somewhat shorter when smaller diameter test bars are employed. Results from such a series of tests at 1200 F. on carbon-molybdenum steel test specimens of varied diameters have been reported.[12] These tests indicated, as would be expected, that the largest change in rupture time occurred between tests on specimens smaller than $\frac{1}{4}$ in. in diameter. With larger diameter specimens, the effect of oxidation was less noticeable. This indicates, as Mr. Jasper suggests, that with much larger sections, the accelerating effect of intergranular oxidation on the fracture time would undoubtedly be less important. There are cases, however, as with boiler tubing, where the wall thickness is even less than that of the ordinary test specimen. For such uses the present test results should be directly applicable.

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[10] Consulting Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[11] Metallurgical Engineer, Research Laboratory, General Electric Co., Schenectady, N. Y.

The author agrees with Messrs. Clark, White, and Fleischmann, that the occurrence of an intergranular type of fracture must not necessarily be associated with low values of hot ductility. Indications are, however, that with the so-called high creep strength low-alloy steels, very low values of elongation are found from long-time tests in the lower temperature ranges. At the higher temperatures, the intermetallic structures which produce the high creep and rupture strengths at the lower temperatures do not remain stable. Due to spheroidization and graphitization, resistance of the grains to deformation by shear is greatly decreased, and increased hot ductility at fracture generally results.

Messrs. Clark, White, and Fleischmann have apparently interpreted the statement that the presence of 4 or 5 per cent of chromium is necessary to insure against intergranular cracking to mean that the addition of 4 or 5 per cent of chromium alone is all that is necessary. This was not the implied meaning. Actually, the statement reads "It appears that an addition of 4 or 5 per cent of chromium is necessary in any ferrous alloy, regardless of its chromium equivalent composition, if complete freedom from intergranular cracking is to be insured." To this probably should have been added that providing sufficient ferrite-forming elements are added to produce an alloy having a chromium equivalent composition in excess of +9, at least 4 or 5 per cent of chromium must also be present.

The author agrees that the outer limits of the closed two-phase borderline field are not too well established, and that with some alloys, this complex field may extend to values considerably greater than the +13 value as stated. However, as far as behavior under stress at elevated temperatures is concerned, the present limits suffice to define the fields with reference to their susceptibility to failure by intergranular cracking in the 900 to 1300 F. temperature range.

Mr. McCutcheon has presented some very interesting results in relation to service records on the 1 per cent chromium, 0.50 per cent molybdenum alloy at 1000 and 1100 F. The author agrees that this alloy should be very desirable for service at 1000 F. In the annealed condition, good values of elongation will result from rupture tests of reasonable duration. However, long-time fractures at 1000 F. or 1100 F. with this alloy will be of the intergranular type even though the elongation is still ample. The object of this paper was not to condemn any alloy, but merely to point out what can be expected as regarding fracture type with the various alloy compositions.

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[12] R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures After Prolonged Loading," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 135, pp. 559–582 (1939).

The statement by Mr. McCutcheon that intergranular cracking does not mean that the material has become embrittled cannot be over-emphasized. Intergranular cracking, as the author understands it, is merely a mechanism of failure and has nothing at all to do with the phenomenon of brittleness as we commonly speak of it with reference to cast iron. In fact, some of the high-chromium ferritic type alloys which are known to develop extreme room temperature brittleness after long-time exposures at elevated temperatures are completely immune to failure by intergranular cracking. Thus, it would seem desirable to speak of the "brittle" appearing intergranular fractures as having low hot ductility rather than as being "brittle."

Mr. Miller's results with carbon molybdenum steel serve to emphasize the point that with a given material, the structure which gives the highest creep and rupture strength also gives the lowest values of hot ductility at fracture. The effect of higher testing temperatures, as we know, with the low-alloy steels is to alter their structure in much the same manner as would a heat treatment at a higher drawing temperature. Mr. Miller's results from step-up tests on carbon-molybdenum steel at 1100 F. are entirely reasonable. The effect of increasing the stress to such a value that failure results in a short period of time would logically be to increase the amount of elongation occurring within the grains themselves. In this sense, the problem of the intercrystalline *versus* the transcrystalline type fractures can be thought of as one of crystal mechanics. If the total resolved shearing stresses on the polycrystalline grains are sufficient to produce continued deformation, ductile type fractures will always result. With lower stresses, if the contrary is true, deformation will occur mostly along the grain boundaries and intergranular type fractures must result. Since grain boundary deformation can only occur with low rates of deformation, the possibility that some of our commercial materials may have greater ductility in service from conditions of over-heating or over stressing is, as Mr. Miller points out, entirely reasonable. Probably the constant stress rupture test will, in tests of long duration, produce the least ductile fractures that can be obtained in tension tests of a given material at elevated temperatures. However, many types of service duplicate this low, constant stress condition, and this should not be overlooked.

Mr. Nadai's question as to whether a vacuum creep test has been made and whether the presence of air influences creep can be only partially answered. We have made creep and rupture tests on pure metals and alloys both in vacuum and in hydrogen, and as far as the type of fracture resulting is concerned, find no difference from the tests in air. However, where oxidation in air is a factor, the creep strength and rupture time are greater when the tests are made in vacuum or in hydrogen. From all of our work, indications are that intergranular oxidation has the effect of accelerating the fracture time, once intergranular cracking has started. However, the phenomenon of intergranular cracking appears to be due to something else besides oxidation.

The question of test bar size was also asked by Mr. Jasper, and that discussion will also serve to answer Mr. Nadai.

HIGH TEMPERATURE RUPTURE AND CREEP TESTS

By Ernest L. Robinson[1]

Year-long tension tests on the materials now commonly used in the construction of machinery and equipment to operate at temperatures in the range between 750 and 1000 F. generally break with a small fraction of the elongation which occurs in an ordinary short-time tension test. The indications of such tests are that breaks will occur with even less amounts of extension and at still lower stresses after still longer periods of time.

Although there have been several papers[2] published on this subject during the past few years, the results of such tests are not yet available in such bulk as to form a generally recognized basis for design. Furthermore, the importance and significance of such tests are not fully understood. It is the object of this paper to discuss the relations of such tests to creep tests and to point out the importance of accumulating more such information.

Rupture Tests

When a series of bars is tested at high temperature and at successively lower stresses, rupture occurs after longer and longer periods of time at smaller and smaller extensions. When the stresses and the extensions at rupture are plotted on log-log paper against the time for rupture, after the first few hundred hours a straight line relationship frequently encourages the extrapolation of the results across another square of the logarithmic coordinates so that, whether really justified or not, rather convincing conclusions are arrived at.

Figure 1 illustrates at the top the method of plotting the test results to give the 100,000-hr. rupture strength. The break in the line, which occurs in this case at approximately 500 hr., is a well-known characteristic. However uncertain the extrapolation may be, it is based on tests five times as long as the 1000-hr. creep tests sanctioned by the A.S.T.M.-A.S.M.E. 1938 Creep Data Book.[3] In any case it is hard to imagine that the material could stand a greater load for 100,000 hr. than that indicated. The extrapolation of the line in the lower portion of Fig. 1 for elongation at rupture is not so convincing. However, exact values of deformation are not so important as exact values of stress. On the other hand, this final branch of the line represents uniform elongation in a long specimen rather than the necking down that occurs after brief periods of stretching in the conventional 2-in. test specimen.

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[1] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

[2] For example:

A. E. White, C. L. Clark and R. L. Wilson, "The Fracture of Carbon Steels at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. XXV, September, 1937, pp. 863-888.

A. E. White, C. L. Clark and R. L. Wilson, "The Rupture Strength of Steels at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. XXVI, March, 1938, pp. 52 80.

R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures After Prolonged Loading," *Metals Technology*, April, 1939, Technical Publication No. 1034 (18 pp.).

R. H. Thielemann, "Some Effects of Composition and Heat Treatment on the High Temperature Rupture Properties of Ferrous Alloys," *Preprint No. 46*, for 1940 Annual Meeting, Am. Soc. Testing Mats., June 24-28.

[3] Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys, published jointly by the American Society for Testing Materials and The American Society of Mechanical Engineers (1938). (Available as a separate publication.)

CREEP TESTS

For machinery that cannot be allowed to deform or distort more than a small fraction of a single per cent throughout its life, it is important to have creep test information. It is not the object of this paper to discuss the various ways of running creep tests, but only to mention certain relations between creep tests and rupture tests. The measurement of extension during the progress of rupture tests is desirable.

One of the more common ways of running creep tests and one recognized in the A.S.T.M.-A.S.M.E. 1938 Creep Data Book is to pull a series of bars at successively different stresses for a period of 1000 hr. and then to plot the resulting final creep rates on log-log paper against the stresses which cause them. A nominal strength is given by the stress which causes a final rate of 0.01 per cent per 1000 hr. (It is common to say "1 per cent in 100,000 hr." but this wording leads to confusion with the stress which would cause a total extension of 1 per cent at the end of 100,000 hr. which may be much greater or less depending on the material and the temperature.) The degree of extrapolation implied in the interpretation of ordinary creep tests can be visualized by noting that each dash of the broken lines in Figs. 2 to 7 is approximately 2000 hr. long.

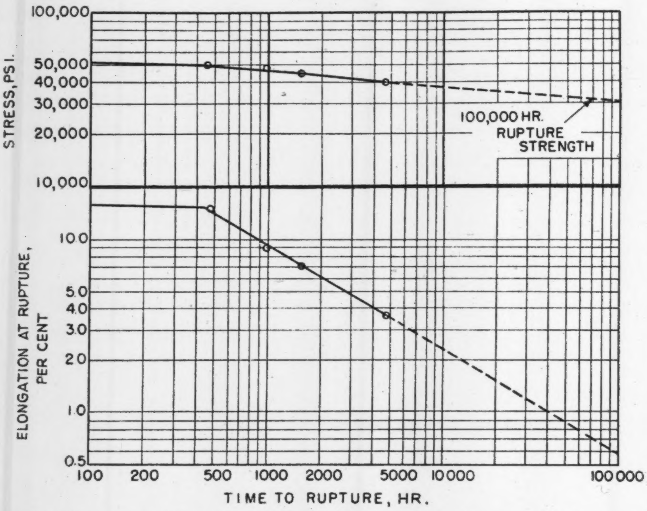

FIG. 1.—Rupture Tests at 900 F., Normalized carbon-molybdenum pipe.

RUPTURE TESTS AND CREEP TESTS

The stress which extrapolation indicates will cause rupture after 100,000 hr., is frequently about double the stress which will cause a creep rate of 0.01 per cent per 1000 hr. at the end of a 1000-hr. test, but materials which have what is called "high creep strength" appear likely to rupture at 100,000 hr. with only 1 per cent extension. On the other hand, materials which have low creep strength appear to have definite margins of extra strength against rupture. The 100,000-hr. rupture strength as indicated by year-long tests may be as great as 4 to 6 times the

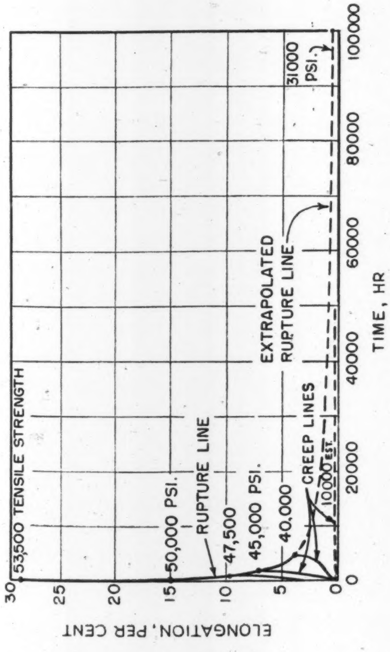

FIG. 2.—Long-Time Performance at 900 F. Normalized carbon-molybdenum pipe.

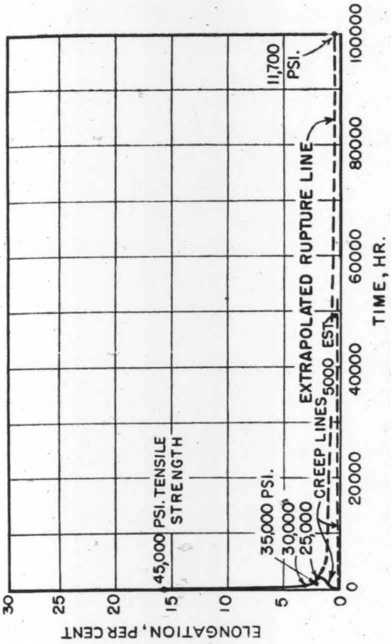

FIG. 3.—Long-Time Performance at 1000 F. Normalized carbon-molybdenum pipe.

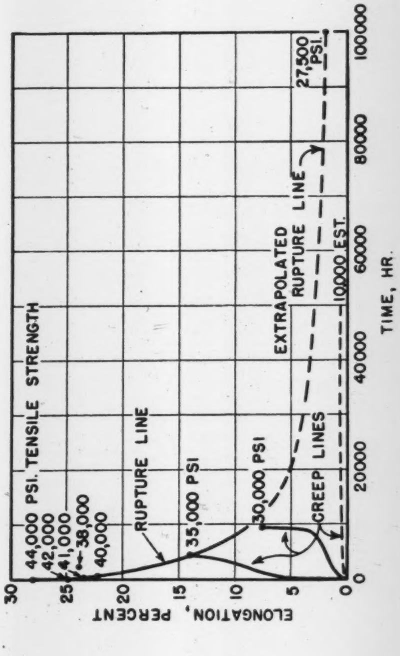

FIG. 4.—Long-Time Performance at 900 F. Annealed carbon-molybdenum pipe.

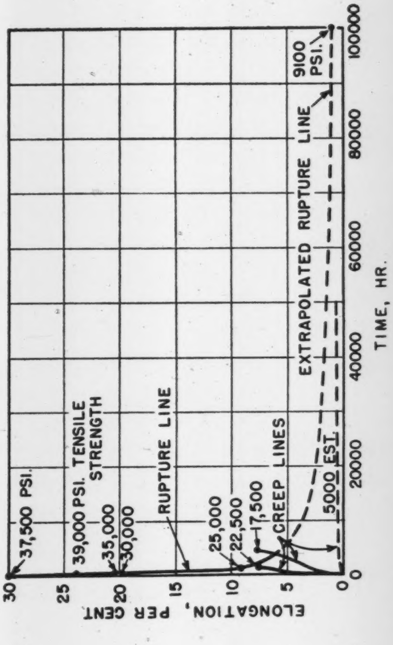

FIG. 5.—Long-Time Performance at 1000 F. Annealed carbon-molybdenum pipe.

nominal creep strength for a rate of 0.01 per cent per 1000 hr.

The diagrams shown in Figs. 2 to 7 are plotted in ordinary scales in order to present to the eye the relations between rupture strength and extension due to creep. The full lines represent tests actually carried to completion. The broken lines have been sketched in under the guidance of logarithmic plottings similar to those illustrated in Fig. 1 for the construction of Fig. 2. At best, these illustrations must be regarded as representing estimated performance subject to revisions which may be important when more information is available. However, such revisions are more likely to be downward rather than upward. In other words, it does not seem possible that longer tests can give much greater strengths than shown, but they might indicate the onset of serious weakening not yet displayed and, furthermore, 10-yr. breaks might conceivably occur with extensions much smaller than the very small elongations shown. It does not seem possible for long-time breaks to have greater elongations than the minimum so far found in test.

Carbon Molybdenum Steel:

Figure 2 shows the behavior at 900 F. of low-carbon 0.5 per cent molybdenum steel in the normalized condition. While this diagram leaves much to be desired in the matter of completeness, the 100,000-hr. rupture strength appears to be three times the 10,000 psi. working stress frequently authorized for steam pipes and drums. However, the test results shown seem to preclude the possibility of more than 2 or 3 per cent of stretch in any long-time failure, and indicate the probability of less than 1 per cent after 100,000 hr.

Figure 3 illustrates the same material at 1000 F. In this case the 100,000-hr. rupture strength seems to have dropped to little more than double the working stress value. Although only a few results are available, breaks of 2 and 3 per cent elongation have occurred in a few months and it seems likely that long-time breaks can have little if any more than 1 per cent elongation and perhaps much less.

Figure 4 represents the same material in the annealed condition at 900 F. The strength is slightly reduced as compared with the normalized material, but the early breaks occur with much greater amounts of extension. Thus, although by no means certain, the possibility of annealed material having greater extension at 100,000 hr. than the stronger normalized material is not precluded by the results available. It must be kept in mind that the stress to cause rupture in 100,000 hr. seems to be much better known than the amount of creep that precedes the break.

Figure 5 shows annealed carbon-molybdenum steel at 1000 F. Although this is the weakest condition among these four illustrations, the probable elongations at long-time rupture appear less than in Fig. 4.

Comparison of these four illustrations shows that when the strength is reduced by use at a higher temperature, the elongation at rupture is also reduced, but when the strength is reduced by annealing, the elongation is increased. Of course longer time tests will give more definite information as to the exact amounts of elongation to be expected at rupture after long service.

Silicon-Chromium-Molybdenum Steel:

Figures 6 and 7 represent the behavior at 1100 and 1200 F., respectively, of a variety of steel containing 1.5 per cent silicon, 5 per cent chromium, and 0.5 per cent molybdenum.

This steel at these higher temperatures

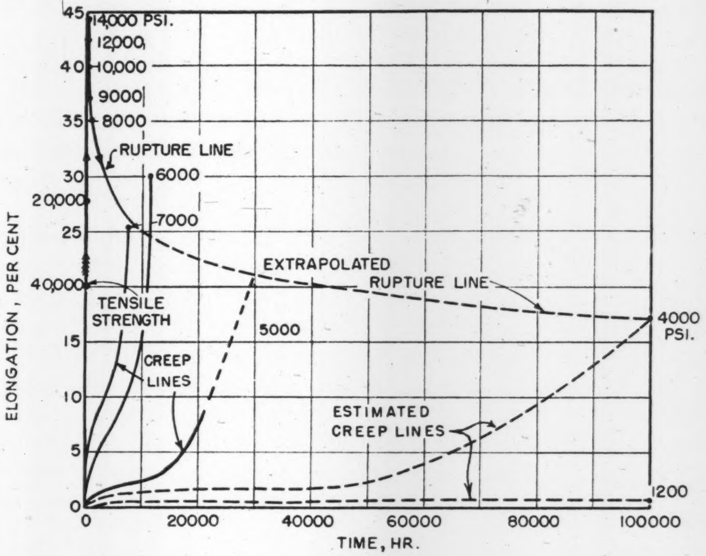

FIG. 6.—Long-Time Performance at 1100 F. 1.5 per cent silicon, 5.0 per cent chromium, 0.5 per cent molybdenum tubing.

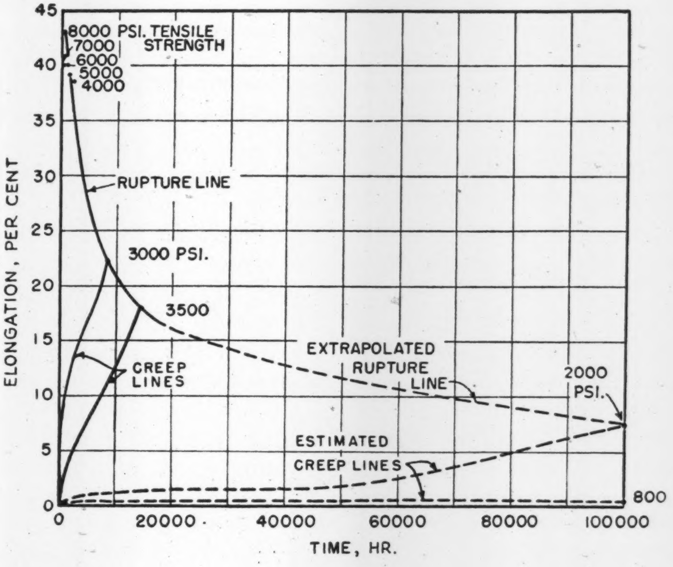

FIG. 7.—Long-Time Performance at 1200 F. 1.5 per cent silicon, 5.0 per cent chromium, 0.5 per cent molybdenum tubing.

has lower rupture strengths than the carbon-molybdenum steel in the more moderate range, but the extensions that accompany rupture are greater. The uncertainty as to how to draw the extension curve out to 100,000 hr. becomes more evident to the eye. Extrapolated creep tests indicate much smaller rates of extension than implied by extrapolation of the extensions measured at rupture. Consequently, a curved dotted line has been drawn which will probably need correction in the future. However, the important matter is the noteworthy difference from the other material. These tests do not preclude the possibility of finding desirably large extensions after years of stress-carrying service.

CONCLUSIONS

Consideration of these test results leads to several questions which seem difficult to answer without years of actual service including service severe enough to indicate what type of distress must be expected. It seems plain that materials put their best foot forward and hang together better for a year or two than for much longer periods of time. Where working stresses correspond to a creep rate of 0.01 per cent per 1000 hr., it seems likely that a year or two of successful service can be easily misinterpreted to give a false sense of success and security. On the other hand, where it is important to limit deformation as in steam turbines, working stresses are more likely to be chosen to correspond with a rate of 0.001 per cent per 1000 hr. Steam turbines have to be designed with both sets of limitations in mind and are thus safeguarded with what we believe to be adequate margins of safety.

The early stages of creep must tend to iron out stress concentrations, and this would seem to be a beneficial process. Elastic concentrations in good design rarely multiply applied loadings by more than 3 to 1 and even with high stresses at moderately high temperatures such concentrations imply extensions in the order of $\frac{1}{4}$ per cent. It would seem reasonable to conclude that creep would go a long way to wipe these out and no harm done.

On the other hand, it is at the moderate temperatures where strengths are high that rupture occurs with the least extension. Frequently there is a degree of follow-up elasticity so that relief of the concentration might not be satisfied by a stretch of $\frac{1}{4}$ per cent. Under such circumstances, it is a fair question whether it is safe to assume that the integrity of the material will be preserved or whether incipient cracks will be started increasing susceptibility to fatigue and corrosion.

At lower temperatures the tensile strength divided by a working stress gives a "factor of safety" which may be in the order of 4 or 5 depending on service conditions. If creep strengths are chosen with reference to a rate of 0.001 per cent per 1000 hr. as with turbines, the 100,000-hr. rupture strength may be 4 or 5 times the creep strength. But if creep strength is chosen with reference to 0.01 per cent per 1000 hr., the apparent long-time factor of safety may be much less.

Actually when treatments are varied the rupture strength is affected much less than the creep strength. Thus if working stresses are set with reference to a low creep strength material, the rupture strength may be 4 times the working stress. On the other hand, if the treatment is varied to improve the creep strength, the rupture strength may turn out to be no more than double the nominal creep strength and some tests show the margin to disappear entirely.

Where the amount of creep is not in

itself an important consideration, there is good reason for design to be guided by rupture strength rather than by creep strength. On the other hand, where the amount of extension, distortion, or deflection is of prime importance both creep strength and rupture strength must be considered.

TABLE OF MATERIALS

CARBON-MOLYBDENUM STEEL

Source: Cut from 6-in. diameter pipe.
Composition: Carbon 0.14; molybdenum 0.46; manganese 0.44; silicon 0.16 per cent.
Physical Properties at Room Temperature:

	Normalized	Annealed
Tensile strength, psi	61 600	56 800
Elastic limit, psi	40 000	31 000
Elongation, per cent	35.5	44.4
Reduction of area, per cent	67.6	68.9

High-Temperature Properties:

		900 F.	1000 F.
Figs. 2 and 3. Normalized from rolls (without draw)	Creep strength 0.01 per cent per 1000 hr.	30 000	7 000
	100,000-hr. rupture strength	31 000	11 700
Figs. 4 and 5. Annealed 8 hr. at 1650 F. and furnace cooled	Creep strength 0.01 per cent per 1000 hr.	13 000	3 000
	100,000-hr. rupture strength	27 500	9 100

SILICON-CHROMIUM-MOLYBDENUM STEEL

Source: Cut from $\frac{3}{4}$ by $1\frac{1}{8}$ in. tubing as manufactured.
Composition: Carbon 0.13; silicon 1.5; chromium 4.98; molybdenum 0.50; manganese 0.25 per cent.
Physical Properties at Room Temperature:

Tensile Strength, psi	78 200
Elongation, per cent	46.5

High-Temperature Properties:

	1100 F.	1200 F.
Creep Strength 0.01 per cent per 1000 hr., psi	1600 to 2800	800 to 1100
100,000-hr. rupture strength, psi	4000	2000

DISCUSSION

MR. SABIN CROCKER[1] (*presented in written form*).—Mr. Robinson's paper is timely in that it surveys a field in which the effect of high temperature on certain metals is suspected to be disquieting, but about which too little is known to be sure of the grounds for concern. In examining fractured creep test specimens, several investigators have observed that a number of the alloys commonly used because of their superior creep resistance are prone to fail by separation of the crystals under prolonged loading at high temperatures. Such failure is attended by only a fraction of the elongation which would occur on a short-time test.

The tendency for alloy steels to fail in this manner was accorded considerable notice by British engineers before it was taken up with serious concern in this country. In an article published in 1932 Rosenhain[2] pointed out that as far back as 1913 this phenomenon had been observed and reported by J. C. W. Humfrey and himself. Mr. Rosenhain noted the tendency of creep specimens to fail by intercrystalline separation and warned against confusing such failure with the embrittlement which occurs in certain types of bolting materials. In 1935 Bailey[3] suggested guarding against this type of failure by limiting the total creep to an extension of 0.5 per cent, or less, over the life of the particular equipment. More recently Tapsell[4] concluded that failure of molybdenum steels by intercrystalline cracking will not occur until at least one per cent deformation has taken place.

As the author points out, specific information on this subject is meager despite its antiquity and the number of writers who have discussed it. Hence the author has been confined to giving results for only two steels (1) a 0.5 per cent molybdenum and (2) a 1.5 per cent silicon, 5 per cent chromium, 0.5 per cent molybdenum steel. It may be of interest, therefore, to point out that good ductility is retained by certain alloys with much lower chromium content than 5 per cent. A 1.4 per cent silicon, 1.24 per cent chromium, 0.58 per cent molybdenum tube in an experimental 1100 F. steam line at the Trenton Channel[5] power plant of The Detroit Edison Co. increased in diameter $\frac{3}{8}$ in. or an increase of 7 per cent without any evidence of intergranular failure or brittleness, although the test was not carried to failure. The operating stress varied from 6780 to 8420 psi. during the progress of 8000-hr. service at 1100 F. Thinning of the tube wall from creep accompanied by air and steam corrosion was responsible for the change in stress. This material, because of an extra addition of silicon, was less resistant to creep and steam corrosion than a variety having only 0.78 per cent silicon, yet it apparently was much stronger than the

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[1] Senior Engineer, Engineering Division, The Detroit Edison Co., Detroit, Mich.

[2] Walter Rosenhain, "Quick Determination of Limiting Creep Stress," *Metal Progress*, February, 1932, pp. 65 66.

[3] R. W. Bailey, "The Utilization of Creep Test Data in Engineering Design," *Proceedings*, Inst. Mechanical Eng., Vol. 131, pp. 294, 300 (1935); also *Engineering*, Vol. 140, pp. 595, 647 (1935).

[4] H. J. Tapsell, "Creep at High Temperatures," *Engineering*, March 15, 1940, pp. 287 288.

[5] R. M. Van Duzer, Jr., and Arthur McCutchan, "High Temperature Steam Experience at Detroit," *Transactions*, Am. Soc. Mechanical Engrs., July, 1939, p. 395.

5 per cent chromium, 0.5 per cent molybdenum pipe reported by the author.

Similar tests on carbon-molybdenum tubes at 925 F. under a stress of 9220 psi., which have progressed for 12,000 hr., also should be of interest. A diametral extension of 0.4 per cent has been measured on one of these thin-walled tubes. Possibly, if a total extension of 1 per cent is obtained in these tests it will give a service check on liability of carbon-molybdenum steels to fracture with such an extension.

The author observes that *normalized* carbon-molybdenum steel has better creep resistance than the *annealed* material, but surmises the relations may be reversed with respect to the elongation attending failure and points out the need for longer time tests to establish this possibility fully. In attempting to understand the underlying reasons for this difference in behavior, the writer would like to point out the possible significance of differences in the respective grain structures produced by normalizing and annealing coarse grained carbon-molybdenum pipe material.

In the normalized condition this material has a grain structure closely resembling that of the hot-finished pipe in the as-rolled condition. The pearlite and excess ferrite are intermingled forming what is termed Widmanstaetten areas which are surrounded by ferrite grains. In the annealed condition this material has a grain structure in which the pearlite exists as discrete patches and the excess ferrite is completely coalesced, forming large equiaxed grains of pure ferrite. The usual photomicrograph at 100 diameters shows the respective pearlite or ferrite grains as all black or all white, while the Widmanstaetten areas show alternate black and white striations throughout each area.

Laboratory creep tests of 8000 hr. duration at 925 F. and a stress of 9250 psi. have been made at the University of Michigan on samples of carbon-molybdenum steel exhibiting a Widmanstaetten and a typical annealed structure. It would seem that less possibility of eventual failure exists in the case of the Widmanstaetten structure since the reported rate of creep for that structure was zero as against 0.5 per cent in 100,000 hr. for the annealed structure.

One point that warrants investigation concerns whether material susceptible to intergranular failure can be detected by removing samples from articles in service and subjecting them to some sort of test. From Mr. Rosenhain's article[2] it would appear that if a specimen could be tested at room temperature just prior to its failure under creep loading, it would not disclose brittleness. To put the question in another way, is there any method of detecting approaching failure of a pipe line by removing samples every few years to check any possible tendency to intergranular failure of a brittle nature? If so, the writer would appreciate advice on how to go about it.

MR. H. L. BURGHOFF[6] (*presented in written form*).—Creep tests appear to have been given much more consideration than rupture tests by most laboratories. It is obvious that the one test supplements the other and that both yield information valuable to design engineers. It would seem that accurate knowledge of creep rates is desirable even in cases where allowable deformation may be great, for the user of equipment will always want to be aware of the course of change in its dimensions. Regardless of creep rate, the possibility of sudden and unexpected failure should not be overlooked and it is in this respect that rupture tests appear most valuable. As the author suggests, the existence of stress concentrations which cannot be calculated may render material susceptible to unforeseen failure. If there is some advance knowledge of the degree of deformation that should result from such a condition, confidence in service behavior is enhanced. If there is likelihood of sudden failure without measurable deformation, the user will at least be somewhat prepared.

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[6] Research Metallurgist, Chase Brass and Copper Co., Waterbury, Conn.

With regard to creep tests of 1000 hr. duration, such as reported so frequently in the A.S.T.M.-A.S.M.E. 1938 Creep Data volume, it is our experience that creep rates of copper alloys based upon such testing periods are frequently distinctly different from rates determined from tests of 5000-hr. duration, which is our normal creep testing period. The rates indicated at the shorter period are higher on the whole and hence lead to more conservative application of material. However, we have encountered some instances in which the creep curves enter the third or final stage, that of increasing creep rate, after somewhat more than 1000 hr. The value of a testing period as long as is feasible is therefore apparent.

MR. H. F. MOORE.[7]—As has been the case in considering creep so now in studying tensile strength and ductility, analogies between the behavior of steel at elevated temperatures and lead alloys at low temperatures appear. Some leads show remarkable loss of elongation after fracture under long-continued steady load. One lead showed 40 per cent in 1 in. for a short-time (testing-machine) load and 1 per cent under a load of some months.

The tensile strength of lead alloys under long-time steady load may be plotted as a straight line on log-log or on semi-log paper with time as (log) abscissas. A few tests have shown some evidence of "flattening out" of the tensile strength - time diagram (like an *S-N* fatigue diagram) but not enough yet to justify conclusions.

MR. M. B. HIGGINS.[8]—I am one of those engineers who have to use creep data for design; in fact I have been designing high-temperature vessels for twenty years, and in the light of the information available now, we were in bad shape twenty years ago. However, none of the vessels has failed and we were using a factor of safety of four, coupled with inspection. What we want is more interpretation of these tests, both the creep and the stress rupture tests, for conversion into working stresses. If there is a way to interpret these data, which engineers are not fully competent to do, I should like to see it done by some one.

MR. ERNEST L. ROBINSON[9] (*Author's closure*).—Mr. Crocker calls attention to certain papers on the subject of brittle fractures antedating those cited by the author. Although there was no intention to present anything like a complete bibliography, the list selected began purposely with the 1937 paper by White, Clark, and Wilson. The metallurgical explanations of the short breaks offered by previous writers reminded the author of Lord Kelvin's remark to the British Institute of Civil Engineers on May 3, 1883:

"I often say that when you can measure what you are speaking about, and express it in numbers, you know something about it; but when you cannot measure it, when you cannot express it in numbers, your knowledge is of a meager and unsatisfactory kind; it may be the beginning of knowledge, but you have scarcely, in your thought, advanced to the stage of Science, whatever the matter may be."[10]

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[7] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[8] The Texas Company, New York, N. Y.

[9] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

[10] Popular Lectures and Addresses by Sir William Thomson, "Electrical Units of Measurement," Vol. 1, p. 80, Macmillan and Co., London and New York (1891).

When C. L. Clark plotted *Stress* against *Life* on log-log paper and showed that the resulting straight line enabled the definite prediction of long-time performance, high-temperature rupture testing attained the status of a science and became capable of application by engineers.

The various contributions of data from the experiences of the Detroit Edison Co. are valuable and welcome. More such information is needed.

Mr. Crocker's final question is important. As far as the author knows, it is not now possible to detect the onset of any weakening until rupture is about to occur, and yet the regularity of behavior indicates that physical effects must be taking place. Doubtless more will be known about this in the future.

Mr. Burghoff has mentioned an important point in the superiority of 5000-hr. creep tests over 1000-hr. tests, although the author agrees with him that the shorter test is usually conservative as a creep test. In any case, as Mr. Burghoff states, information is needed on the rupture strength.

Mr. Moore's hint that the long-time tension tests on lead alloys show occasional evidence of a true rupture limit below which failure will not occur no matter how long the service, is very encouraging. However, regardless of how much we may hope for such a conclusion, it seems necessary for the present to go by the indicated relationships until there is a sufficient accumulation of very long-time tests covering periods of years to demonstrate the reality of such a limiting rupture strength.

The author joins Mr. Higgins in his desire for more significant interpretation of these data. The results suggest that the 4 to 1 factor of safety mentioned by Mr. Higgins was wisely chosen years ago. For that very reason it does not seem to the author that there is anything to show that the designs were in bad shape then. It is evident that the true factor of safety has been less than anticipated. Under such circumstances the accumulation of definite experience can easily justify the use of smaller factors of safety with reference to new knowledge, provided, of course, the experience is analyzed as systematically as test results, and presented in justification of the proposed action. There is no more valuable guide for the designs of the future than the experience of the past.

In concluding, the author desires to note that Figs. 4 and 5 have been revised since the issuance of the preprint. The 30,000-psi. bar in Fig. 4 broke shortly after the original completion of the manuscript with a life of 9027 hr. and an extension of 7.5 per cent, both smaller than anticipated, and it seemed necessary to revise the extrapolation downward. Figure 5 was similarly altered without any further data at 1000 F. The final point at 900 F. was just one more point lessening the uncertainty that might possibly have permitted the hope of a little larger elongation in connection with a long-time test.

HIGH-SPEED TENSION TESTS AT ELEVATED TEMPERATURES

By M. Manjoine[1] and A. Nadai[1]

Synopsis

Very little is known about the actual forces required to deform copper, steel, or some of the other ductile metals at very high velocities of strain and at elevated temperatures. A high-speed tension testing machine was developed in which the tension diagrams of metals were recorded. The test specimens occupied the axis of a copper coil which acted as an induction furnace, in which temperatures up to 1200 C. could be reached. The tension tests were carried out at an approximately constant relative velocity between the heads. Stress-strain curves were automatically recorded on the screen of a cathode-ray oscillograph, using the elastic strain in a very rigid steel bar as a means for the indication of the load and the relative movement of the heads of the specimens for determining the strains. A series of very rapid tension tests with soft annealed copper at elevated temperatures is described, while the results of tests with steel and other metals will be reported elsewhere. The general trend of the speed law for plastic flow of copper is indicated.

The continuous hot and cold strip mills built during the last 10 to 15 yr. in this country stand out as one of the most remarkable developments in the mass production of steel. In these continuous mills, wide sheets of steel are rolled under high velocities. Rolling speeds of 1000 to 1800 ft. per min. and more are used. Very little is known about the forces required for the pure plastic forming of steel and of other ductile metals at the high straining velocities used for rolling of sheets or bars or for drawing of rods and wires from atmospheric temperature up to forging temperatures.

Assuming a draft of 50 per cent in one single pass of a sheet, this would correspond to an elongation of 100 per cent or to a conventional strain $\epsilon = 1$ in the direction of rolling and to a conventional unit shear $\sigma = \tan (90° - 28°) = \tan 62° = 1.88$. Assuming further an entering speed of 10 ft. per sec. and a leaving speed of 20 ft. per sec. or an average speed of $v = 15$ ft. per sec. = 180 in. per sec. of the sheet, a diameter of the rolls of 20 in. and a thickness of the sheet equal to 1 in. on the entering and of $\frac{1}{2}$ in. on the leaving side, the time for covering the horizontal width ($x = 2.24$ in.) of the pressure arc along the rolls corresponding to this reduction is of the order of $t = x/v = 2.24 \div 180 = 0.0124$ sec. The mean rate of stretching of the sheet in the direction of rolling is therefore equal to $\epsilon/t = 1 \div 0.0124 = 80$ per second. Deformation or strain rates of the order of 100 to 400 per second are practically realized in these rapid forming processes, although in many cases the true linear rates of stretching in rolling or drawing may be somewhat less. It was thought that it would be of considerable general interest to investigate the laws of deformation of some of the ductile metals under these conditions up to the range of the hot-forming (forging) temperatures.[2] In the high-speed tests to be reported below the strain rates varied from 100 to 1000 per second. The fastest rates in these tests were two to three times larger than the fastest rates encountered in rolling or drawing operations.

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[1] Mechanical Engineer, and Consulting Mechanical Engineer, respectively, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

It is perhaps instructive to compare with these fast rates the other extreme range of deformation rates in which engineers are also quite interested in certain engineering applications. In steam turbines, machine parts are subjected to prolonged loading and to continuous heat and must be designed so that very slow rates of deformation are prescribed. For example, the permissible "creep" may be one-tenth of 1 per cent strain in 100,000 hr. (corresponding to 11 yr. service). This would correspond to a "creep rate" of 10^{-8} per hour or to a strain rate = 2.8 times 10^{-12} per second. The fastest rate of the deformation which was mentioned above is, therefore, 3.6×10^{14} times the slow rate.

These figures may illustrate the enormous range of the order of 10^{14} over which the rate of strain may vary in different applications in engineering practice. The authors do not know of other engineering problems in which one of the physical variables would have to be considered over such a wide range of its possible values.[3] An ordinary "short-time tension test" in which a bar may be stretched 50 per cent in $\frac{1}{4}$ hr. corresponds to a strain rate $0.5 \div 0.25 = 2$ per hour or $2 \div 3600 = 0.00056$ per second.

For testing the behavior of metals at high velocities, originally torsion tests were planned with hollow specimens of a comparatively short length. Preliminary tests, however, indicated that it was difficult to prevent plastic buckling of the short tubes and the torsion tests were abandoned in favor of rapid tension tests.

The behavior of the ductile metals under fairly rapidly increasing or impact loads has been the subject of many investigations. In the latter part of the nineteenth century the notched-bar tests in bending were introduced for studying the resistance of the metals to impact and since then a large engineering literature has developed on this subject. References with regard to impact testing in bending and concerning the notched-bar tests will not be considered in this paper since the efforts of engineers and metallurgists to develop the notched-bar test as a means for testing heat treatment, notch sensitivity, and other more or less complex mechanical properties of the ductile metals have recently been treated at various occasions.[4]

It was thought that it would be valuable to review, briefly, first the more recent attempts of several investigators to describe the plastic behavior of the ductile metals under simple states of stress such as pure tension or compression when the velocity of the plastic deformation is increased. The fact that the forces, under which the ductile metals yield continuously, increase in general quite considerably if the speed of the deformation increases, did not escape the attention of the early investigators of the mechanical behavior of the metals and numerous references to it can be found in the older handbooks on testing of materials associated with the names of Hopkinson, Duguet, Tresca, de St. Venant, Martens and others, and in the handbooks on metals which were published during the last two or three decades of the nineteenth century. In connection with these attempts perhaps the name of Ludwik[5] deserves to be mentioned especially, because he clearly included the influence of the speed of plastic deformation in his considerations by stating that the internal resistance of a metal to a permanent change of shape (at normal temperature) increases both with the permanent strains and also with the rates with which these latter are changing. In his tension test experiments with tin wires he found that the ultimate stress increases as the logarithm of the rate of strain.[6] As far as the yield stress at a given strain is concerned, two dissertations (Cassebaum and Deutler[7]) influenced by Prandtl[8] arrived at the same conclusion. The latter in his mechanical model of a solid body includes for a certain range of the plastic rates of strain the logarithmic speed law as a general expression connecting the rates of strain with the yield stresses.

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[2] Plans for a survey of plastic deformation at extremely high rates of strain were submitted in 1937 to the Engineering Foundation in New York and to several companies. This work was sponsored by the A.S.M.E. Research Committee on Rolling of Steel and carried out under a grant received from the Engineering Foundation and with the financial assistance of United Engineering and Foundry Co. in Pittsburgh and the Westinghouse Research Laboratories, East Pittsburgh.

[3] In hydrodynamics the Reynolds number determines the type of flow and the range of this fundamental variable is of the order of 10^7.

[4] Concerning impact testing, reference is made to the two symposiums on impact testing reported in *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, pp. 5–149 (1922); Vol. 38, Part II, pp. 21–156 (1938).

For studying the influence of the speeds of plastic deformation upon the yield stresses, "dynamic" stress-strain curves observed in rapid tension or compression tests were compared with the "static" diagram taken under ordinary testing velocities. Two methods have been used: an indirect and a direct method. In the first the length l of a tension test specimen is observed as a function of the time t while the bar is stretched by the falling weight or a vertical drop weight or a pendulum impact machine. From the curve $l = f(t)$ the retardation d^2l/dt^2 of the falling weight during the tension test is computed. By multiplying the mass of the weight with the retardation d^2l/dt^2 the loads are found under which the bar is stretched. This indirect method seems to have been first applied by Plank.[9] It requires a double differentiation of an empirically given curve and for this reason cannot be considered as a convenient or sufficiently exact method for obtaining stress-strain curves in rapid tests. In the second method for obtaining stress-strain curves the loads are directly observed.

Koerber and Storp[10] found for four carbon steels that the ultimate strength in their "dynamic" tensile stress-strain curves was 1.15 to 1.42 times larger and their elongation at rupture 1.05 to 1.12 times larger than the corresponding quantities in the static tests. The maximum load was reached at a smaller amount of deformation than in the slow tests. The average rate of plastic strain in their tests was of the order of 100 per second.

Meyer[11] stretched thin wires by a weight, which was not allowed to drop from a higher position, but was released at the beginning of the test while already attached to the wires. The strain rates in these tests were a good deal smaller than those obtained by Plank or Koerber and Storp, and of the order of 5.0 per second. Correspondingly, he found a smaller speed effect for mild steel, brass, and copper (tested in the annealed and hard condition) and only an increase by a factor of 1.06 to 1.12 for the ultimate strength and even a decrease of the elongation at rupture.

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[5] P. Ludwik, "Elemente der technologischen Mechanik," pp. 44, 47 Julius Springer, Berlin (1909).

[6] *Physikalische Zeitschrift*, Vol. 10, No. 12, p. 411 (1909).

[7] Cassebaum and Deutler, Doctor Dissertations, Goettingen (1911 and 1932).

[8] L. Prandtl "Ein Gedankenmodell zur kinetischen Theorie der festen Koerper," *Zeitschrift* für angewandte Mathematik und Mechanik, p. 85 (1928).

[9] R. Plank, "Betrachtungen über dynamische Zugbeanspruchung," *Zeitschrift* des Vereines Deutscher Ingenieure, Vol. 56, pp. 17–24, 46 51 (1912).

[10] F. Koerber and H. Storp, "Über den Kraftverlauf bei der Schlagprüfung," *Mitteilungen* des Kaiser Wilhelm Instituts für Eisenforschung, Düsseldorf, Vol. 7, pp. 81 97 (1925). Compare also F. Koerber and R. H. Sack Vol. 4, p. 11 (1922) in the same journal.

[11] Eugen Meyer, "Der Verlauf des Zugversuches bei raschem Zerreissen," *Forschungsarbeiten* des Vereines deutscher Ingenieure, No. 295 (Carl von Bach commemorative volume), pp. 62–73 (1927).

Brinkmann[12] ran a few impact tests with copper wires by recording the load with piezo crystals and found agreement between his tests and some compression tests made by Siebel and Pomp[13] regarding the influence of speed, for which he assumes the logarithmic law.

Honegger[14] computed the loads in his tension impact tests by a double differentiation of the recorded relative motion between the two heads of the test specimens. The velocity of the moving pendulum weight decreased in his tests during the process of the impact in the proportion 14:3. The initial rate of strain was of the order of 100 to 160 per second. For soft aluminum and for soft copper, respectively, he found the tensile strengths 1.22 to 1.27 and 1.19 to 1.24 times and the elongations at rupture 1.18 to 1.27 and 1.11 to 1.25 times, larger than in the static tests. For hard copper or aluminum the increases were less. The energy for breaking these metals at room temperature was 38 to 52 per cent larger for soft aluminum and 30 to 35 per cent larger for soft copper than in the static tests.

A valuable series of tension tests in which the effect of the speed of deformation clearly showed up is the one reported by Weerts.[15] Using the experimental setup of Meyer he observed the stress-strain curves of 27 single crystal bars of pure aluminum in tension tests, of which 14 were tested dynamically, and 13 in slow tests. The gage length of the aluminum single-crystal bars was 100 mm., their diameter 3.4 mm.; the crystals were broken in 0.1 sec. The bars broke at about 70 per cent elongation; the mean rate of strain of the rapid tests was of the order of 7 per second. It should be noted that test results were always compared for single crystals having a similar orientation of their axes in their initial positions relative to the direction of pull. Weerts found that the ultimate tensile stress in the dynamic tests was 18 per cent higher than for the static tests.

In the preceding investigations either the Charpy pendulum or the drop weight type of impact machines were utilized. A rotary type of impact machine was introduced in France by Guillery[16] in which the kinetic energy of a flywheel is used for breaking the specimens.[17]

A rotary impact machine in which tension tests at considerably larger rates of stretching were run than any of the rates which were previously reached, was recently described by Mann.[18] Steels and bronzes were tested up to maximum rates of strain of the order of 3840 per second. Mann observed the impact energies (not the full diagrams) and found a "transition" velocity or tension rate of stretching characteristic of each metal, above which the impact energy would decrease rapidly with increasing strain rates. This transition strain rate he found at 1540 and 720 per second for an S.A.E. 1035 steel in the quenched-and-drawn and in the annealed condition, respectively. For an S.A.E. 2340 steel the corresponding rates were 2160 and 600, respectively.

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[12] H. Brinkmann, "Zerreissversuche mit hohen Geschwindigkeiten," Dissertation, Hannover, 32 pp. (1933).

[13] E. Siebel and A. Pomp, "Zur Weiterentwickelung des Druckversuches," *Mitteilungen* des Kaiser Wilhelm Instituts für Eisenforschung, Düsseldorf, Vol. 10, p. 55 (1928); "Einfluss der Formaenderungsgeschwindigkeit auf den Verlauf der Fliesskurve von Metallen, Vol. 10, p. 63 (1928).

[14] E. Honegger, "Schlagzerreissversuche an Aluminium und Kupfer," *Bericht 95*, Eidgenössische Materialprüfungsanstalt an der E.T.H., Zürich, pp. 1–11, April, 1935.

[15] J. Weerts, "Dynamische und statische Zugversuche an Aluminium Einzelkrystallen," Dissertation, Berlin (1928).

[16] Guillery, *Engineering*, January 12, 1906, p. 49. A brief description with views of the Guillery tester may be found in the Book by A. Martens and E. Heyn, "Handbuch der Materialienkunde," Part 2, Vol. A, pp. 380, 381, Julius Springer, Berlin (1912). A view of a later type of this machine also in G. Charpy and A. Cornu-Thenard (Paris), "New Experiments on Shock Tests and on the Determination of Resilience," *The Journal of the Iron and Steel Institute*, Vol. XCVI, No. 11, pp. 61–119 (see Fig. 7 on p. 87 (1917)).

[17] The wheel can be rotated by hand or by a motor. The notched bar is mounted on a movable support. With the wheel an iron mass is revolving in the path of which the bar has to be brought by a rapid motion. The impact energy is recorded by means of a hydraulic tachometer on which the drop of the speed of the wheel can be read. The rates of strain which could be reached in the Guillery impact machine were perhaps of the order of 200 per second.

Moore[19] used the elastic extension of a steel bar to measure the load on a tension test specimen which was rapidly loaded by a falling weight.

A few high-speed tension stress-strain curves have been recorded by D. W. Ginns[20] in a machine, in which a large elastic spring served for loading. Forces were measured by means of a substance (carbon) whose electric resistance varied with the pressure exerted on it. The rates of strain were of the order of 60 per second.

Rapid tension tests were also reported by Clark and Dätwyler[21] who used a steel bar of rectangular cross-section around which was wound a fine constantan wire the electric resistance of which changed with the applied force in the steel bar. The rates of strain they observed were of the order of 150 per second. They found the ultimate stress 30 to 40 per cent higher than in the static tests and observed in a few cases a decrease of the impact energy in dynamic tests.

One of the most extensive studies on the speed of deformation for metallic materials at low and at high temperatures which came to the attention of the authors is the series of papers on torsion impact tests by Mititosi Itihara.[22] He used a somewhat unusual shape of specimen having a constricted portion (8 mm. in diameter and 10 mm. long) in a cylindrical bar of 20-mm. diameter with an over-all length of 120 mm. The torques were measured by means of a spring. The deflection of the spring and the angles of twist were recorded by optical means on revolving drums as functions of the time. Rates of shear of 100 per second could be produced in the short gage length and differences of 25 per cent between the torques in the dynamic and the static tests were observed at atmospheric temperature. The largest effects were found at the beginning of the stress-strain curves. The angle of twist at rupture was 10 to 20 per cent larger in the rapid torsion tests. Mild steel, ingot iron, and a 0.3 per cent carbon steel were investigated in an extended series of torsion tests at temperatures from -150 to 1000 C. Itihara found that the blue-brittleness range shifted to a higher temperature and the maximum torque increased when the velocity of deformation was increased. In general the shape of his torque-twist curves had a great resemblance to the tension test diagrams described below.

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[18] H. C. Mann, "High-Velocity Tension-Impact Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 85 (1936). This machine is capable of producing a peripheral velocity of 1000 ft. per sec. or a rate of strain of 12,000 per second in a 1 in. long specimen. The impact energy is observed by means of recording the angular motion of a pendulum mounted coaxially with the flywheel.

[19] H. F. Moore, "Measurement of the Force of Impact by Means of the Elastic Stretch of a Steel Bar," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 124 (1922).

[20] The mechanical properties of some metals and alloys broken at ultra-high speeds. D. W. Ginns, *Journal*, Inst. Metals, Vol. LXI, p. 61 (1937).

[21] D. S. Clark and G. Dätwyler, "Stress-Strain Relations Under Tension Impact Loading," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 98 (1938).

[22] "Impact Torsion Tests," *Technology Reports*, Tohoku Imperial Univ., Senday, Japan: First part, Vol. 11, pp. 16-72 (1933); Second part, Vol. 11, pp. 73-95 (1934); Third part, Vol. 11, pp. 97-111 (1934); Fourth part, Vol. 11, pp. 112-165 (1934); Fifth part, Vol. 12, pp. 63-104 (1935); Sixth part, Vol. 12, pp. 105-118 (1935).

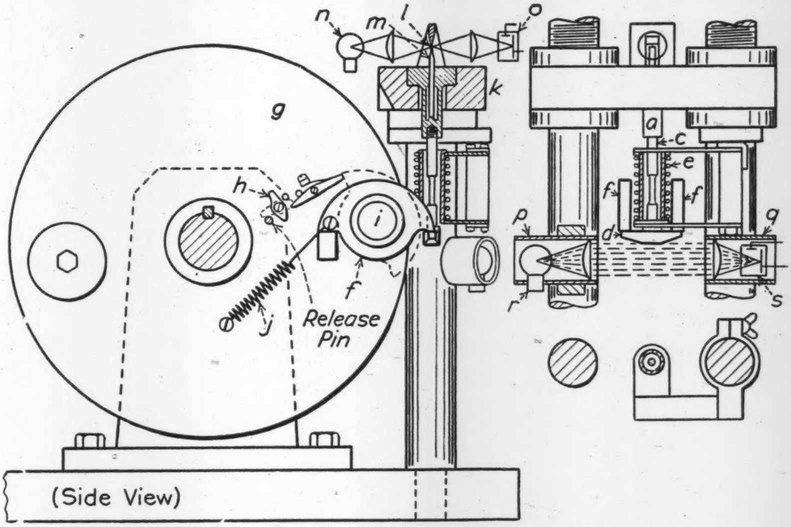
n
m
l
o
k
g
h
i
f
j
Release Pin
a
c
e
f
f
p
d
q
r
s
(Side View)

FIG. 1.—High-Speed Tension Test Machine.

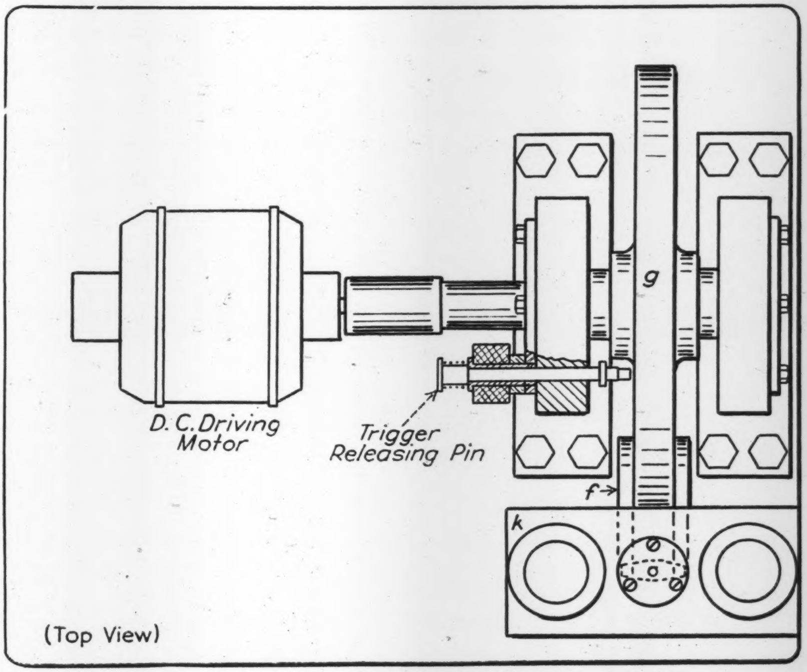
g
D. C. Driving Motor
Trigger Releasing Pin
f
k
a
(Top View)

FIG. 2.—High-Speed Tension Test Machine (Top View).

A certain amount of criticism must be voiced concerning the use of such a short constricted portion as the one mentioned above in the gage length of Itihara's test specimens. The ratio of the length to the diameter was 1.25. In such short specimens the distribution of stress in a torsion test must have been considerably disturbed or different from the plastic distribution of stress in a long cylinder; the shearing stress *versus* unit shear curves could hardly be predicted from the observed torque-twist diagrams.

The High-Speed Tension Test Machine:

The machine shown in the Figs. 1 to 4 consists of a flywheel driven by a d.c. motor, a pair of hammers rotating with the wheel, a pair of vertical columns carrying a heavy cross-piece on which the test specimen is mounted in a vertical position, and the force and strain measuring devices. The force measuring bar *a* (see also Figs. 5 and 6) is attached to the upper head *k*. A cooling plug *b* (Figs. 5 and 6) is placed in series with the bar *a* and the test specimen *c*. This latter carries at its lower end an anvil *d*, a short cross-piece of steel which is threaded to admit the test bar (*c*). The specimen is heated by an induction furnace (*e*) consisting of a water-cooled copper tube wound as a helical coil. The force is applied to the specimen through a pair of hammers *f* striking the anvil *d*. The two hammers *f* are pinned to the flywheel *g* by means of a heavy pin *i* around which they can be rotated. When the trigger *h* is released, the hammers are rotated in their striking position by means of the spring *j* which is attached to the flywheel *g*. The trigger is tripped by a solenoid operated pin (Fig. 2).

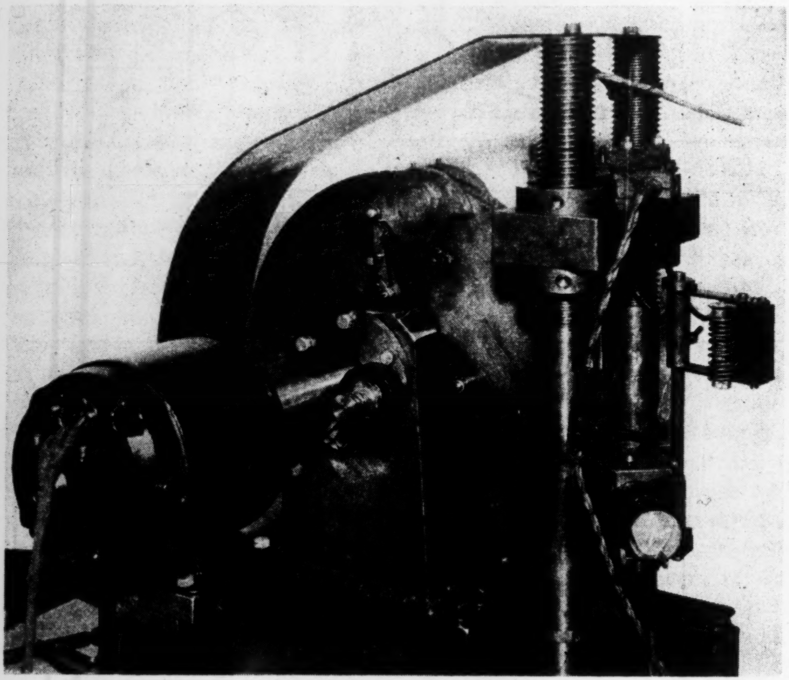

FIG. 3.—High-Speed Tension Test Machine.

When the hammers hit the anvil, a

force is transmitted through the specimen *c* to the force-measuring bar *a*. The extremely small elastic extension of the bar *a* is converted in an electric current impulse which is proportional to the force. The motion of the anvil is utilized for measuring the strain. When the anvil moves vertically downward, it cuts a light beam falling on a photoelectric cell (Fig. 6). The resulting current decrease is proportional to the strain. These two current impulses are combined at right angles on the screen of a standard-type 168 Dumont cathode-ray oscillograph which therefore records the stress-strain curve.

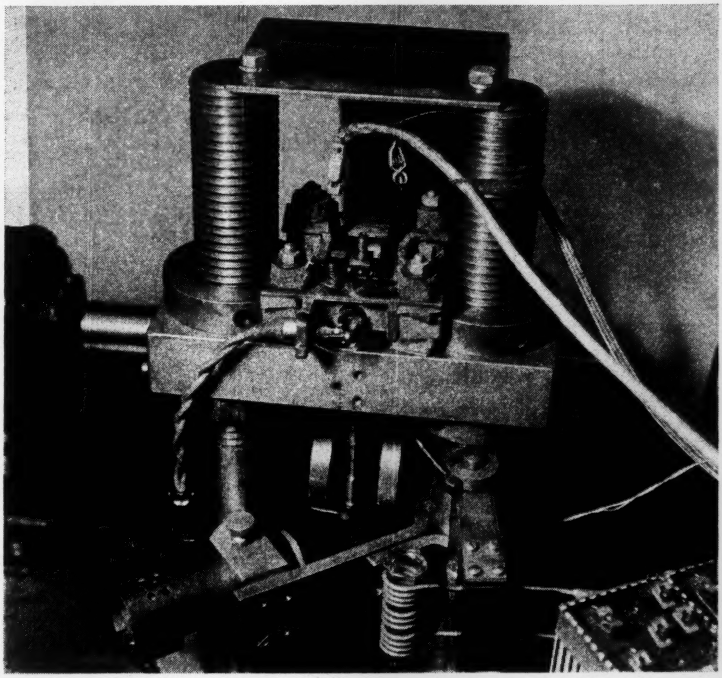

FIG. 4.—High-Speed Tension Test Machine.

The force measuring bar *a* (Fig. 5) is made from a solid piece of tool steel. The two upright sections transmit the load to the upper head and their elastic deformation appears as a small change of the width of a very narrow optical slit *l*. A vertical pin *m* which is welded to the bar *a* transmits the motion of the lower head of this bar to the slit *l* whose upper edge follows the motion of the upper head of the bar *a*. Through the 0.002-in. slit a beam of light is projected which falls on a photoelectric cell *o*.[23] To prevent overheating of the force-measuring bar a water-cooled plug *b* is inserted between this bar and the test specimen *c*.

The natural frequency of the lowest mode of the longitudinal oscillations of the force measuring bar was made as high as possible. This frequency was estimated to be around 15,000 cycles per second. The attachments to this bar were kept as light as possible with the intention of not reducing this frequency too much. The actual stress-strain curves showed that the lowest frequency of the system, consisting of the force-measuring bar, the test specimen, and the anvil, was of the order of 10,000 vibration cycles per second. The duration of the fastest tests was 5 to 6 ten-thousandths of a second so that usually five to six principal vibrations appear in the fastest diagrams. It was noted that the amplitude of these oscillations increases with the speed of plastic deformation and that these disturbing oscillations were damped down fairly rapidly in many of the materials which

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[23] A narrow illuminated slit through which the light falls on a photoelectric cell has been used in an extensometer described by E. Lehr and H. Granacher: "Dehnungsmessgerät mit sehr kleiner Messstrecke," Forschung, VDI, F. p. 66 (1936).

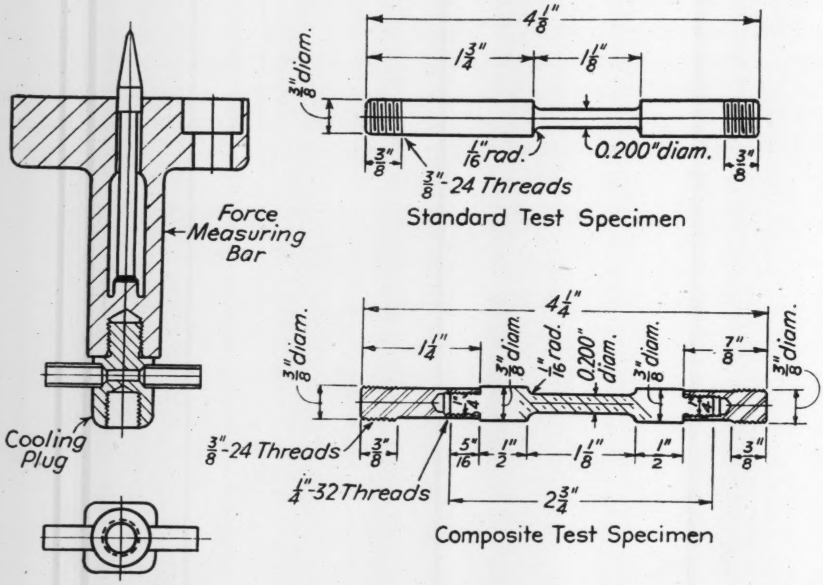

Force
Measuring
Bar
Cooling
Plug
$4\frac{1}{8}''$
$1\frac{3}{4}''$
$1\frac{1}{8}''$
$\frac{3}{8}''$ diam.
$\frac{3}{8}''$
$\frac{1}{16}''$ rad.
0.200" diam.
$\frac{3}{8}''$
$\frac{3}{8}''$-24 Threads
Standard Test Specimen
$4\frac{1}{4}''$
$1\frac{1}{4}''$
$\frac{3}{8}''$ diam.
$\frac{3}{8}''$ diam.
$\frac{1}{16}''$ rad.
0.200" diam.
$\frac{3}{8}''$ diam.
$\frac{7}{8}''$
$\frac{3}{8}''$ diam.
$\frac{3}{8}''$-24 Threads
$\frac{3}{8}''$
$\frac{5}{16}''$
$\frac{1}{2}''$
$1\frac{1}{8}''$
$\frac{1}{2}''$
$\frac{3}{8}''$
$\frac{1}{4}''$-32 Threads
$2\frac{3}{4}''$
Composite Test Specimen

FIG. 5.—Force-Measuring Bar and Test Specimen.

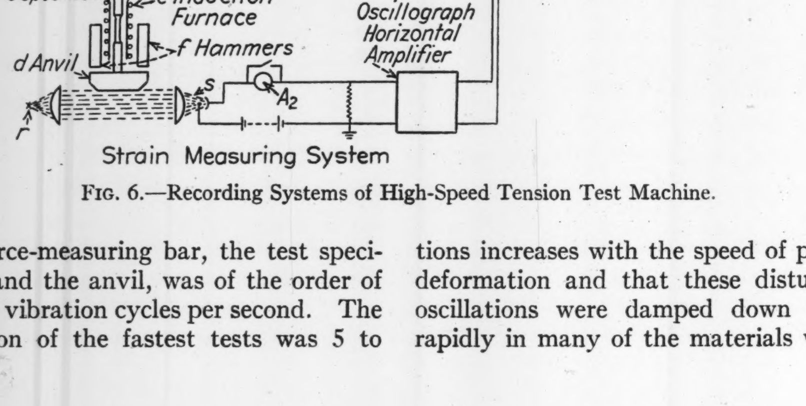
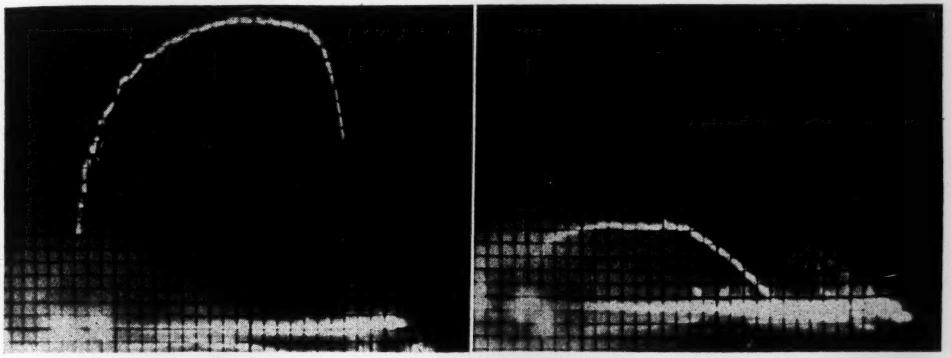

Stress Measuring System
n
l
o
m
k
A_1
D.C. Amplifier
Cathode Ray Tube
Oscillograph
Vertical
Amplifier
Electron
Beam
Camera
a Force
Bar
b Cooling Plug
c Specimen
e Induction
Furnace
f Hammers
d Anvil
s
A_2
r
Oscillograph
Horizontal
Amplifier
Strain Measuring System

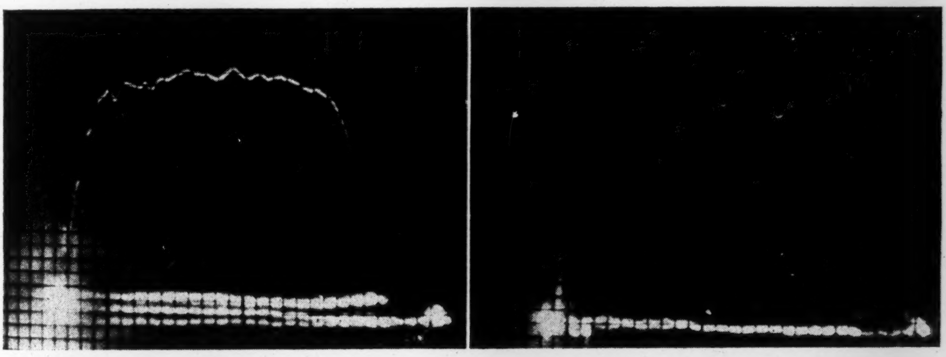
FIG. 6.—Recording Systems of High-Speed Tension Test Machine.

were investigated. At peripheral speeds above 1000 in. per second these oscillations became seriously disturbing and it must be doubted that diagrams could successfully be recorded at much higher velocities in dynamic systems of a similar nature.

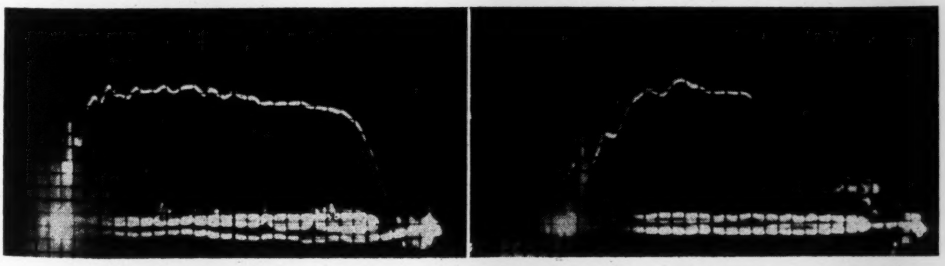


25 C. 1000 C.

Strain rate, 135 per second



400 C. Strain rate, 450 per second. 400 C. Strain rate, 900 per second.



800 C. Strain rate, 450 per second. 800 C. Strain rate, 900 per second.

Fig. 7.—Original Photographs of Copper Tests.

The illumination of the slit for the force measurements is shown in Fig. 6. By means of a lens, the light of a lamp *n* is projected uniformly over the optical slit l. The photoelectric cell o, pickup, and d.c. amplifier are combined into one unit (Fig. 6). An R.C.A. No. 922 cartridge-type of vacuum photoelectric cell was used. The force transmitted through the test specimen stretches the two uprights of the force-measuring bar, the result being that the width of the narrow slit l is slightly increased and more light passes through it to the photoelectric cell o. The response of the photoelectric cell is finally converted by the usual means into a voltage under whose action a cathode-ray deflection is obtained on a screen proportional to the acting force in the test bar.

To insure accurate and reproducible readings, the force-measuring system was calibrated frequently, usually before a series of tests and after it.[24]

For the strain measurements a second optical system with a photoelectric cell was provided, all parts of which were mounted in two coaxial brass tubes, p and q, below the anvil d (Figs. 1 and 6). The light of a small lamp r was converted to a parallel beam of light between two condensing lenses. Only a rectangular portion ($\frac{3}{8}$ by 1 in.) of the parallel beam of light was utilized. When the anvil, due to the stretching of the test specimen, moves downward it intercepts the light and the resulting decrease of light causes a response of the photoelectric cell s which is recorded as a horizontal deflection of the electron beam moving across the screen of the oscillograph. The strain-measuring system was also frequently calibrated. The oscillograms appearing on the screen were photographed on 35-mm. Agfa ultra-speed panchromatic films in a Zeiss Contax camera equipped with a F 1.5 lens.[24]

Test Specimens.—Two types of test specimens were used (Fig. 5)—a standard specimen for tests below 400 C., and a short bar for tests above this temperature. At the higher temperatures the longer bars conducted too much heat to the heads, and a composite specimen was used consisting of a shorter piece of the material to be tested and of two stainless steel heads screwed to the former. Through this arrangement the heat losses through the heads were considerably reduced due to the poorer heat conductivity of the steel and at the same time more heat could be generated in the steel ends from the induction furnace. The diameter of the test section is 0.2 in., the gage length is 1 in. with fillets of $\frac{1}{16}$-in. radius at the shoulders. The effective gage length due to the presence of the two fillets was estimated to be 1.11 in.

Several metals (steel, copper, aluminum, and a very pure iron) were tested. In this report the high-speed tension tests made with copper will be described. The test specimens were machined from a $\frac{3}{8}$-in. diameter round bar of commercially pure copper. The machined specimens were annealed by heating them in dry regenerated gas to 500 C. in 5 hr. After this they were cooled to room temperature in 12 hr.

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[24] The calibration of the force-measuring system is accomplished in the following manner: The grid bias of the first stage tube of the d-c. amplifier is set to give a definite current in the output circuit at meter A_1, Fig. 6, when the light source, n, has been cut off from the optical slit, l. The lamp is now turned on and its intensity increased until the output current has been changed by a certain amount due to the light reaching the photoelectric cell, o, through the no-load opening of the slit, l. When these no-load settings have been made, a load is applied to the force-measuring bar with dead weights acting through a lever arm. The change in the output current is noted for given applied loads. From the resulting current *versus* load curve, the output voltages of the d-c. amplifier can be determined for all loads. The output voltage is put into the oscillograph vertical amplifier which is an a.c. amplifier and must be calibrated dynamically. This is accomplished by applying known a-c. voltages to the amplifier and measuring the resulting beam deflections. By combining the calibrations of the a-c. and d-c. amplifiers an over-all calibration can be determined which gives the screen deflection for any applied load within the range of the calibration. The strain-measuring system is calibrated as follows: The lamp, r, is set at a definite intensity to give a certain current in the photoelectric cell circuit which is measured at the meter (A_2), Fig. 6. A shutter, placed in the position of the anvil, is moved downward a hundredth of an inch and the resulting change in photoelectric cell current recorded. This is repeated until the light beam is entirely cut off. From the data obtained the voltage produced across the photoelectric cell load resistor can be computed. The load-resistor voltage is put into the oscillograph horizontal amplifier which is an a-c. amplifier. This amplifier is calibrated dynamically and in the same manner as the oscillograph vertical amplifier. By combining the calibrations of the photoelectric cell circuit and of the a-c. amplifier, an overall calibration can be computed which gives the horizontal screen deflection for any anvil displacement. In addition, the total stretch of the broken test specimen is measured and compared with photograph record of the test.

Tests with Pure Copper:

Nineteen specimens were tested at the following three rates of strain: 135, 450, and 900 per second corresponding to 166, 556, and 1110 r.p.m. of the flywheel. At each speed tests were run at room temperature and at 200, 400, 600, and 800 C. At a strain rate of 135 per second tests were also made at 900 and 1000 C. The melting temperature of pure copper being at 1083 C., in the last-mentioned test a temperature was reached which was 83 deg. Cent. below the melting point. The stress-strain curves were recorded on 35-mm. film. Tracings of some of the original diagrams are reproduced in Figs. 8, 9, 10, and 11. These curves show the influence of temperature and speed of testing.

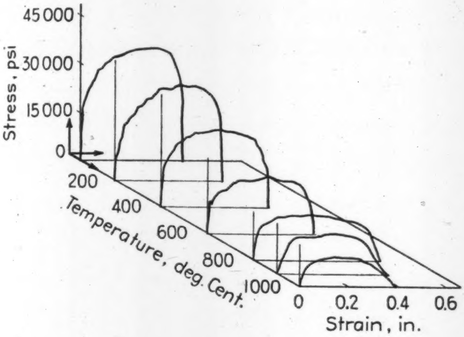

FIG. 8.—Stress-Strain Curves for Pure Copper at Elevated Temperatures, Strain Rate 135 per Second.

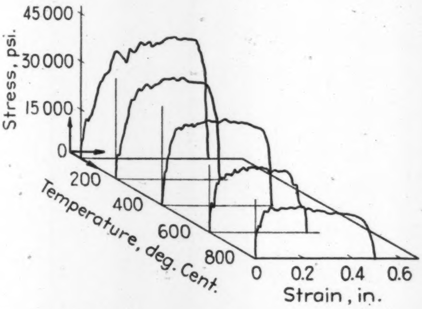

FIG. 9.—Stress-Strain Curves for Pure Copper at Elevated Temperatures, Strain Rate 450 per Second.

Several actual test photographs were selected at random and are shown in Fig. 7. From these diagrams the force-elongation curve can be seen and measured with accuracy. The cross-sectioned grid seen in the photograph is a grid with 0.1-in. divisions which was placed over the end of the cathode-ray screen before the photographs were taken. The large spot at the origin is a fogging of the screen due to the fact that the cathode ray has not yet started its motion. As soon as the force is

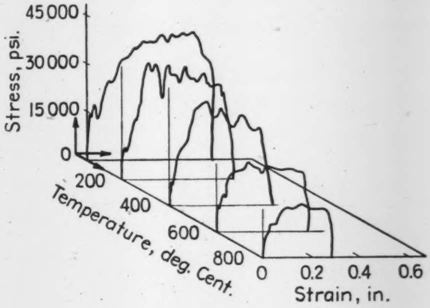

FIG. 10.—Stress-Strain Curves for Pure Copper at Elevated Temperatures, Strain Rate 900 per Second.

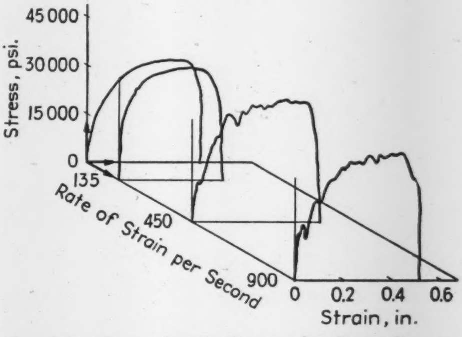

FIG. 11.—Stress-Strain Curves for Pure Copper at High Rates of Strain at Room Temperature.

applied to the specimen the electron beam traces the force-elongation curve on the screen. Several horizontal sweeps of the beam can be seen in addition to the curve. This is due to the fact that the hammers cut the strain-measuring light beam in every revolution of the flywheel. Thus as long as the beam is on and the camera open the horizontal displacement of the beam will be photographed when the strain measuring light beam is intercepted.

TABLE I.—RESULTS OF HIGH-SPEED TENSION TESTS WITH COPPER.

Test	Temperature, deg. Cent.	Strain Rate per sec.	Ultimate Strength, psi.		Strain at Ultimate Strength, per cent	Total Strain, per cent
			σ_0[a]	σ[b]		
No. 1	22	135	37 200	48 000	29.0	48.6
No. 2	28	450	38 700	50 000	29.3	51.8
No. 3	28	900	39 200	51 700	31.8	53.4
No. 4	200	135	32 000	41 100	28.5	47.7
No. 5	200	450	34 000	42 400	24.5	50.2
No. 6	200	900	35 900	43 600	21.1	59.2
No. 7	400	135	25 900	33 000	27.2	51.8
No. 8	400	450	28 500	35 700	25.2	56.3
No. 9	400	900	29 800	36 200	21.2	56.3
No. 10	600	135	19 800	25 200	27.4	48.6
No. 11	600	450	21 500	26 800	24.5	47.7
No. 12	600	900	22 700	27 200	19.4	36.5[c]
No. 12	600	900	22 840	27 400	19.5	33.3[c]
No. 13	800	135	15 400	19 400	26.0	60.3
No. 14	800	450	17 000	20 600	21.0	63.5
No. 15	800	900	18 300	21 000	14.4	25.2[c]
No. 16	900	135	12 620	15 460	22.4	46.8
No. 17	1000	135	9 710	11 280	16.1	42.3
No. 18	25	Static (approx. 5×10^{-4})	30 300	40 300	32.9	47.4

[a] Stress based on original area.
[b] True stress based on actual area.
[c] Broken in the threads.

With reference to the stress-strain curves shown in Figs 8 to 11 it should be noted that the *stress and strain scales are only approximate* but close enough for purposes of comparison. The true values of stress and strain are listed in Table I. Figure 11 shows the effect of speed at room temperature. In Fig. 10 are the curves taken at a strain rate of 900 per second. The disturbances due to the vibration of the force bar system can be seen. The amplitude of this superimposed vibration increases with speed and with the applied force but decreases due to internal damping. The damping effect can also be seen in some of these curves. In Fig. 9, showing the curves taken at a strain rate of 450 per second, the amplitude of the vibration is small and the relative frequency appears higher. The result is only a small disturbance to the curve. At the lowest rate of 135 per second, Fig. 8, the vibration has such a small amplitude and is of such a high relative frequency that practically no effect can be seen in the diagrams. The curves at the high temperatures of 800 and 900 C. show the effect of stronger necking in the specimen. At these high temperatures, the smallest section in the neck is reduced to a point, consequently the load drops nearly to the value zero before the bar breaks. (Compare the photographs of the broken pieces in Fig. 16).

Figure 11 shows a static test besides three high-speed tests. The shape of the high-speed diagrams is nearly the same as that of the static test except that the initial portion of the curves is much steeper. The dependence of the ultimate strength and of the corresponding true stress on the rates of strain is shown in Figs. 12 and 13. In Fig. 14 the same values are plotted against temperature. It is very remarkable that at such high temperatures as 900 or 1000 C. a strong resistance still exists in a metal such as copper when it is deformed at these rapid rates of strain. For example, the ultimate stress at a rate of 900 per second at 1000 C. is approximately one-fifth of its value at room temperature. If one would compare these stresses at considerably lower rates of strain, the decrease at the higher temperatures would have been much greater. Such tests are contemplated for a further comparison. Finally, in Fig. 15 an attempt was made to represent in a semilogarithmic plot the effect of the speed of deformation over a wider range of the rates of strain than those used in the tests mentioned above. In Fig. 15 some points refer to static tests for copper at room temperature, assuming a mean rate of 5×10^{-4} per second. In addition a few points were plotted referring to the fairly rapid test by Brinkmann and Honegger, and also to some slower tests by Siebel and Pomp. The various points seem to agree well with the general trend of an inclined curve which is nearly a straight line. The straight line would correspond in the semilogarithmic plot of Fig. 15 to the

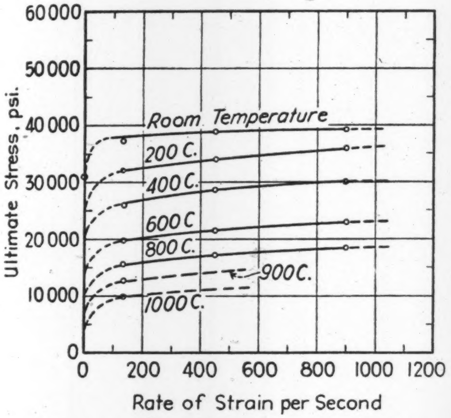

FIG. 12.—Effect of Strain Rate on Ultimate Stress of Pure Copper at Elevated Temperatures.

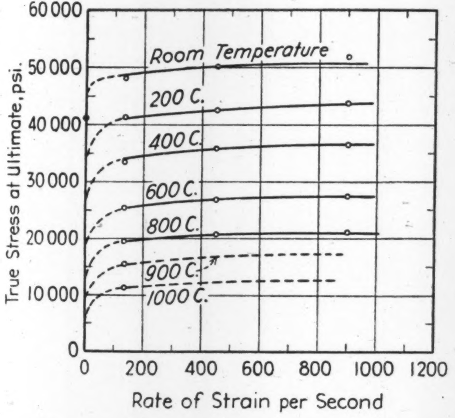

FIG. 13.—Effect of Strain Rate on True Stress at Ultimate of Pure Copper at Various Temperatures.

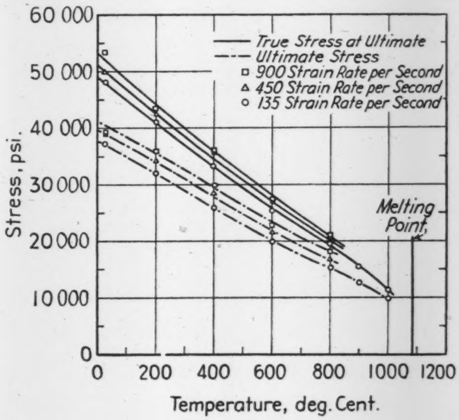

FIG. 14.—Effect of Temperature and Speed on Ultimate Stress for Pure Copper.

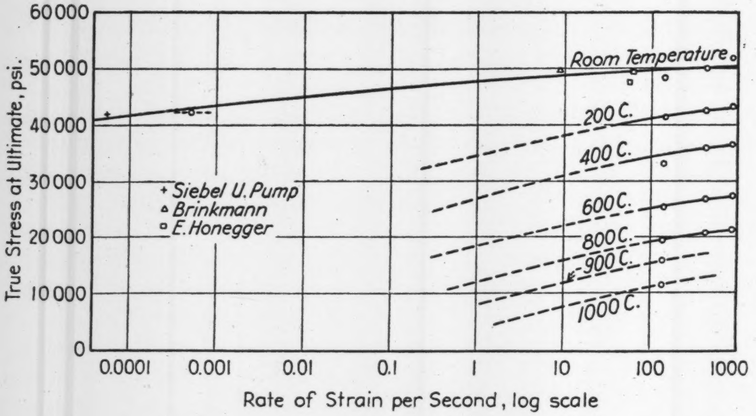

FIG. 15.—Effect of Strain Rate of Pure Copper at Various Temperatures on True Stress at Ultimate Strength.

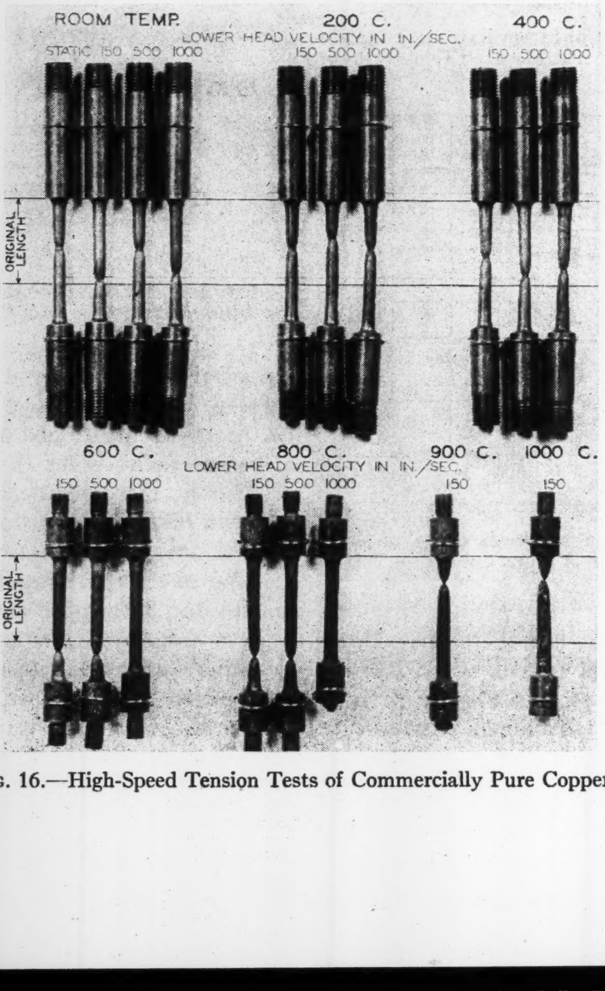
ROOM TEMP.
200 C.
400 C.
LOWER HEAD VELOCITY IN IN./SEC.
STATIC 150 500 1000
150 500 1000
150 500 1000
ORIGINAL LENGTH
600 C.
800 C.
900 C.
1000 C.
LOWER HEAD VELOCITY IN IN./SEC.
150 500 1000
150 500 1000
150
150
ORIGINAL LENGTH

FIG. 16.—High-Speed Tension Tests of Commercially Pure Copper.

logarithmic speed law. This is also in agreement with some tests with various metals which were tested under constant strain rates at elevated temperatures.[25] From the evidence available it seems that the trend of the curve representing the general speed law of plastic deformation at a given temperature is characterized by an inclined straight line in the semilogarithmic plot at the intermediate range of speeds. This curve bends off at both ends of this intermediate range. With a recently built constant strain-rate machine further points in the intermediate range of rates (10^{-5} to 10^{2} per second) are intended to be observed. The high-temperature tests recorded in Table I are also represented by the corresponding points in Fig. 15.

Acknowledgments:

Acknowledgments are due to the American Society of Mechanical Engineers for supporting the plans of this investigation, to the Engineering Foundation in New York for a grant, to United Engineering and Foundry Co. in Pittsburgh for further funds made available, and to the Westinghouse Electric and Manufacturing Co. in East Pittsburgh for the experimental facilities offered in the Research Laboratories, including a high-frequency oscillator. Further thanks are particularly due to O. E. Hovey, Director of the Engineering Foundation, F. C. Biggert, Vice-President of United Engineering and Foundry Co., and E. A. Holbrook, Dean of the University of Pittsburgh for their personal interest in this work, and to L. W. Chubb, Director of the Westinghouse Research Laboratories, for support received from the laboratories.

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[25] "Creep of Metals," *Proceedings*, Internat. Congress Testing Mats., Group A, Metals, London, p. 4 (1937). It should expressly be noted that the points to be compared in Fig. 15 do not refer to the same strain. In a more exact comparison also the strains for which the points are to be compared (in Fig. 15) should be kept constant. In other words, a family of curves should be plotted for each temperature, not a single curve as shown in Fig. 15.

DISCUSSION

Mr. Cyril Stanley Smith.[1]—Can the authors give some additional particulars in regard to the heating of the specimen? In particular it would be desirable to know the speed of heating and the extent of temperature gradients (both radial and longitudinal) existing in the specimen at the time of testing. With rapid high-frequency induction heating such gradients might not be negligible. Where was the thermocouple placed for measuring specimen temperature?

Mr. J. M. Lessells.[2]—With regard to the wavy form of diagram shown by Fig. 7, I should like to ask the authors whether it is their opinion that this is due to the characteristics of the loading or to the material itself. If reference be made to a previous paper by de Forest[3] it will be seen that wavy stress diagrams of a somewhat similar nature are sometimes obtained, but that it was believed this was due to the particular material characteristics. The comments of the authors would be appreciated on this point.

Mr. C. W. MacGregor.[4]—In Table I it is shown that the uniform strain up to the ultimate strength decreases fairly rapidly with increased velocity, especially at the high temperatures. The total strain shows the same effect. Since the material reported herein was copper, I should like to ask whether the same result was found in the other materials tested.

Mention is made of Mann's tests at the Watertown Arsenal in which he gets a transitional velocity. I was wondering whether the authors have also found such a critical velocity. I understand Mann finds a critical velocity in the neighborhood of 700 or so for annealed mild steel, and since the authors' tests come within that range, it would be interesting to know whether they also had found the same effect.

Mr. A. Nadai.[5]—Mr. Lessells asked the question whether we believe that the waviness in some of the recorded stress-strain curves of our rapid tension tests was due to an inherent property of the materials tested or to something else. We are pretty certain that the waves which appear superposed on some of these curves were not caused by properties of the material but were due to vibrations of the mechanical system. Several observations can be quoted to substantiate this statement. If one tries to simplify the mechanical system one may describe it as consisting of an elastically stiff member (the force measuring device) attached to a very large mass (the heavy cross beam of the machine) and supporting some smaller masses (the anvil and the test bar). Mr. Manjoine has made an approximate computation of the lowest natural frequency of this system and the experiments showed that the waves in the diagrams had a frequency of this order of magnitude: namely, about 10,000 cycles per second. Knowing the mean rate of stretching in the recorded diagrams it is easy to estimate the times for one cycle of the oscillations. The faster the rates were the fewer waves appeared in the diagrams but the more the latter appeared disturbed. We drew also the conclusion which we have not expressed in the paper that it is probably difficult or impossible to record such diagrams at much higher speeds than the fastest ones which were used in these tests, because of the increasing dynamic disturbances through the violent oscillations of the mechanical system at still higher velocities.

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[3] A. V. de Forest, "Some Experiments on the Plastic Elongation of Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. XVI, Part II, p. 455 (1916).

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Referring to the question raised by Mr. MacGregor, we have to reply that we never observed a critical velocity in our tests. The following materials were tested: copper, aluminum, pure iron, and mild steel. In not one case have we found a critical speed at which the energy of deformation as represented by the area of the stress-strain curve started to decrease with the increasing speeds of testing. We believe that in mild steel we have probably covered the range of the speeds in which a critical speed should have been observed.

I may mention that Mr. G. F. Jenks made the suggestion that one and the same material should be tested in the machine at Watertown Arsenal and the one in Pittsburgh. We have furnished specimens of copper to Watertown Arsenal with the request that they be tested within the range of strain rates in which a critical speed was to be expected. I had a letter a few days ago stating that no critical speed was found for this copper at the observed rather low velocities. The same was the case for the bars which were tested in Pittsburgh. Soft copper may not have been the proper metal for investigating the question. Some further comparative high-speed tension tests with steels are pending.

MR. M. MANJOINE.[6]—In answering the first question raised by Mr. MacGregor, I can say that in general, for all the materials which were tested, the uniform strain up to the ultimate strength decreased with increasing strain rate whereas the total strain increased. This is also true for copper as shown in Table I. Mr. MacGregor may have been lead to believe that the total strains decreased because at the highest velocity which was tested some of the specimens were unbroken in the gage length and the value of total strain reported refers to the partial strain of the test section.

Answering Mr. Smith, it should be noted that the temperature of the copper specimen was measured by a potentiometer through a thermocouple placed at one end of the test section. From a previous calibration we found that we could obtain a uniform temperature over the entire gage length to within 5 C. by adjusting the field of the induction furnace, that is, by proper spacing of the turns of the furnace coil. No visible effect of a radial gradient of the temperature could be observed because the specimen showed the same type of fracture (cup and cone) for 200 C. as for room temperature. At 600 C. and above the specimens necked down to nearly a point which would be impossible if the surface were hotter than the central portion. This is in accordance with what we might expect of copper which has a high thermal conductivity and for a case in which a larger portion of the heat generated was conducted into the gage length from the heads which are $\frac{3}{8}$ in. in diameter compared to 0.2 in. in diameter at the test section.

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MECHANICAL PROPERTIES OF GRAY CAST IRON IN TORSION

By Jasper O. Draffin,[1] W. Leighton Collins,[1] and Carl H. Casberg[2]

Synopsis

Tests of hollow cylinders of gray cast iron having various ratios of wall thickness to outside radius are reported. Two sizes of cylinders were used, outside diameters of 3½ and 4½ in., with ratios of wall thickness to outside radius (t/r) ranging from 0.443 to 0.057. Tension specimens were cut from various parts of the cylinder wall in a number of unmachined castings in order to furnish additional information on the mechanical properties and uniformity of the cast iron.

The results obtained corroborate those found in earlier torsional tests of small solid and hollow cylinders and show that the modulus of rupture ranges, in a fairly regular curve, from 1.36 times the tensile strength for a solid cylinder to a value of 0.73 times the tensile strength for a hollow cylinder having a ratio of wall thickness to outside radius of 0.057. The curve, if extrapolated to zero t/r, gives a value for the modulus of rupture equal to 63 per cent of the tensile strength.

In 1938 and 1939 the authors presented before the Society papers[3, 4] dealing, among other things, with the torsional properties of ordinary gray cast iron and of high-strength alloy cast iron. In the discussion of the papers it was suggested that information would be desirable over a greater range of sizes, larger diameters, and smaller ratios of wall thickness to outside radius (t/r) than those presented in the papers. Such tests have been made and are herein reported for cylinders having outside diameters of approximately 3½ and 4½ in. and with wall thicknesses ranging from 0.70 in. to 0.10 in.

Material, Test Specimens and Tests

Material:

Twenty hollow circular specimens were cast with square ends. Ten had 3½-in. outside and 1¼-in. inside diameters and ten had 4½-in. outside and 2¾-in. inside diameters. Additional details of the castings are shown in Fig. 3 (*a*). All cylinders were cast in a vertical position, one of each size in a flask, and were poured from the bottom, both from the same sprue. The first pair of cylinders in each heat was marked No. 1 and so on to No. 5, the last pair to be poured in each heat. Both heats of iron were melted in a cupola furnace. Heat No. 1 consisted of 40 per cent each

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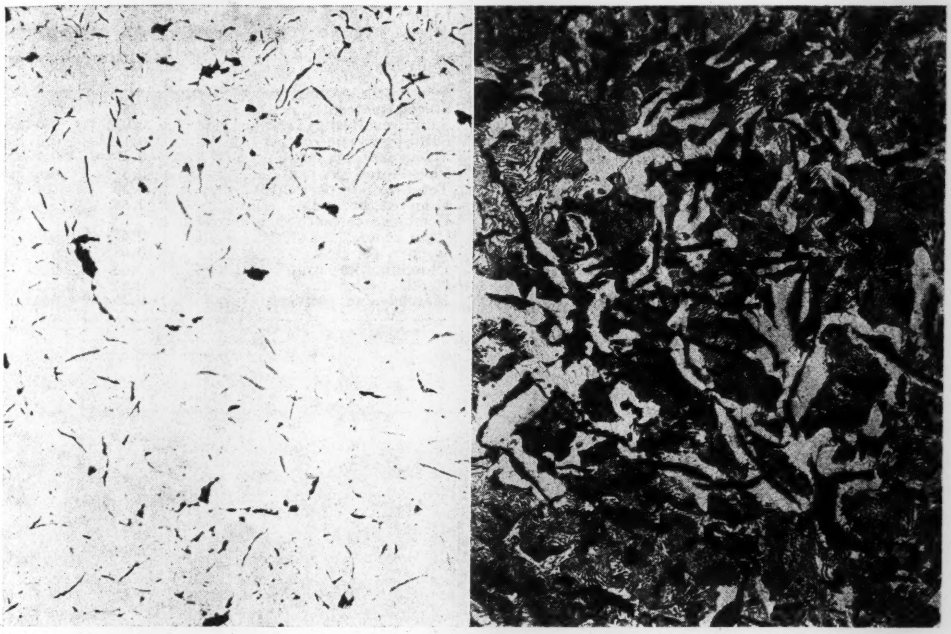
[2] Professor of Mechanical Engineering, University of Illinois, Urbana, Ill.

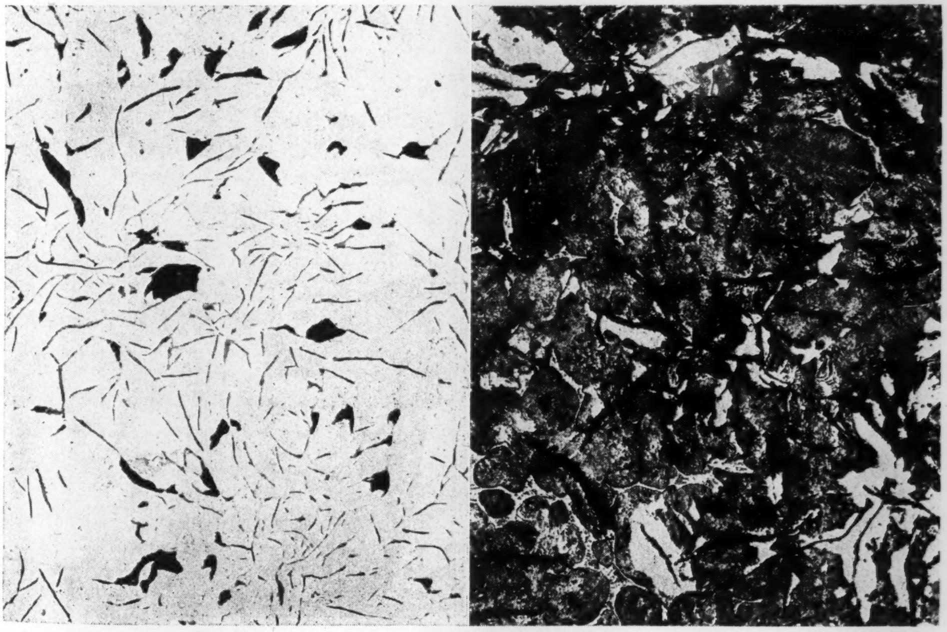
[3] J. O. Draffin and W. L. Collins, "Effect of Size and Type of Specimen on the Torsional Properties of Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 235 (1938).

[4] J. O. Draffin and W. L. Collins, "Mechanical Properties of a High-Strength Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 589 (1939).



(*a*) Unetched (× 50). (*b*) Etched with 2 per cent Nital (× 100).

Heat No. 1, Cylinder No. 2.



(*c*) Unetched (× 50). (*d*) Etched with 2 per cent Nital (× 100).

Heat No. 2, Cylinder No. 2.

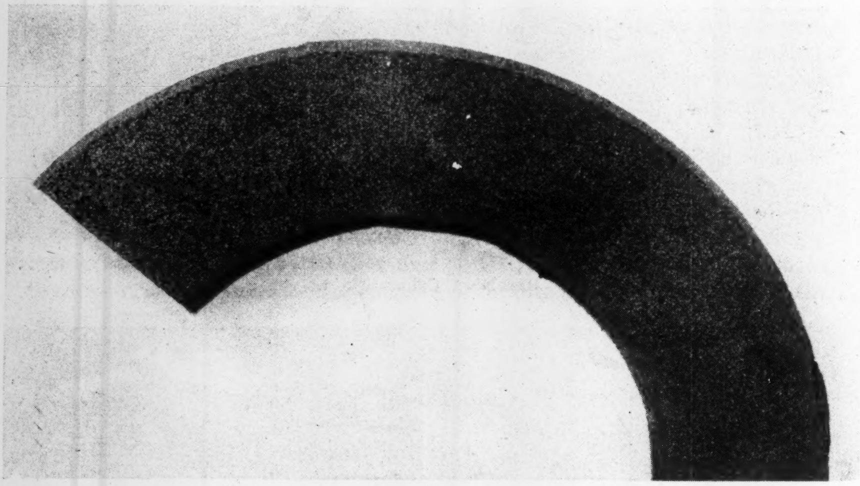
FIG. 1.—Typical Structure of Cast Iron.

of new pig and scrap cast iron and 20 per cent steel scrap, with the addition of small amounts of nickel and molybdenum in the cupola. Heat No. 2 consisted of 50 per cent each of new pig and scrap cast iron.

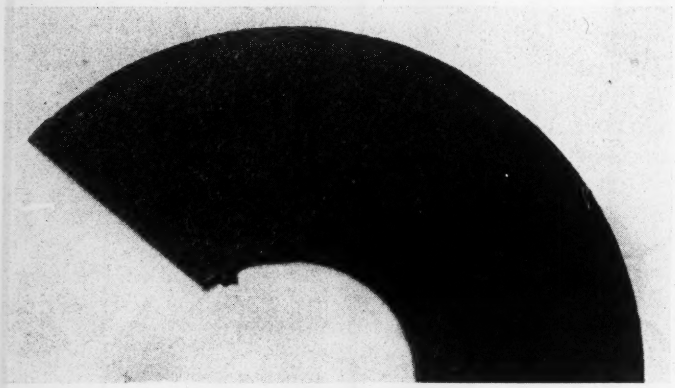
The chemical composition of the two heats, as determined by the American Cast Iron Pipe Co., are as follows. Each value is the average of duplicate analyses except as noted:

	Heat No. 1, Cylinder No. 2	Heat No. 2, Cylinder No. 2
Silicon, per cent	1.84	2.43
Sulfur, evolved as H_2S, per cent	0.099	0.083
Sulfur, gravimetric, per cent	0.119[a]	0.093[a]
Manganese, per cent	0.53[a]	0.99[a]
Phosphorus, per cent	0.52[a]	0.65[a]
Total carbon, per cent	3.20	3.47
Graphite, per cent	2.59	2.68
Combined carbon (by difference), per cent	0.61	0.79
Copper, per cent	0.08[a]	0.07[a]
Chromium, per cent	0.03	0.79
Nickel, per cent	0.21[a]	0.13[a]
Molybdenum, per cent	0.15	0.02

[a] Single analysis.



(*a*) 4½-in. cylinder.



(*b*) 3½-in. cylinder.

FIG. 2.—Typical Structure of Section of Cast Iron Etched with 2 parts H_2SO_4, 1 part HCl and 2 parts H_2O. Natural size.

Photomicrographs of the typical internal structure of both heats are shown in Fig. 1, and etched sections, natural size, are shown in Fig. 2. The location in the castings from which the specimens for metallographic study were taken is shown in Fig. 3 (*c*).

Specimens:

From the castings, see Fig. 3 (*a*), specimens were machined according to the sketches shown in Fig. 3 (*b*). The inside diameters were machined to approximately 1.75 in. and 3.25 in., respectively, for the 3½-in. and 4½-in. diameter castings, the differences in the wall thickness of the different specimens being obtained by varying the outside diameters; this method of obtaining various wall thickness enabled the material at the inside of all the specimens to be at the same distance from the inside of the castings and was used because of the limitations on the equipment available for reaming the interior of the cylinders. Consequently the outer surfaces of the various torsion specimens were at different distances from the outer surface of the casting. In order to determine whether any variation, and if so how much, existed between the outer and inner parts of the wall of the casting and in order to correlate the data from the different heats, small tension specimens, $\frac{3}{8}$ in. in diameter, were cut from the walls of unmachined castings in such positions that the center of one small specimen was at approximately the same location in the cross-section of the casting as the center of the wall thickness of each torsion specimen. The locations of these tension specimens and their dimensions are shown in Fig. 3 (*c*) and (*d*).

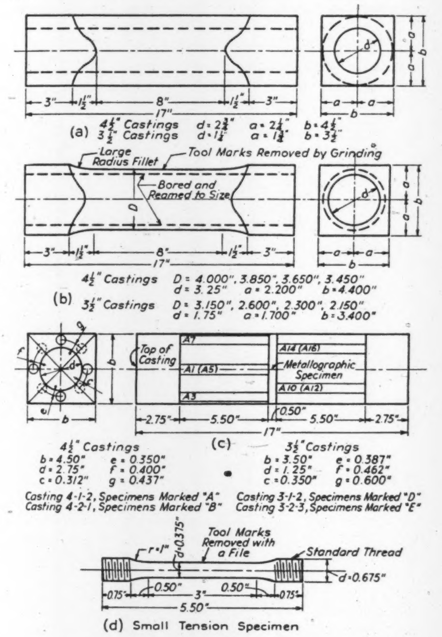

3"
1½"
8"
1½"
3"
17"
a
a
b
d
(a) 4½" Castings d = 2¾" a = 2¼" b = 4½"
3½" Castings d = 1¼" a = 1¾" b = 3½"
Large Radius Fillet
Tool Marks Removed by Grinding
Bored and Reamed to Size
D
(b) 4½" Castings D = 4.000", 3.850", 3.650", 3.450" d = 3.25" a = 2.200" b = 4.400"
3½" Castings D = 3.150", 2.600", 2.300", 2.150" d = 1.75" a = 1.700" b = 3.400"
Top of Casting
A7
A1 (A5)
A3
A14 (A16)
Metallographic Specimen
A10 (A12)
0.50"
2.75"
5.50"
5.50"
2.75"
17"
f
g
e
(c)
4½" Castings
b = 4.50" e = 0.350"
d = 2.75" f = 0.400"
c = 0.312" g = 0.437"
3½" Castings
b = 3.50" e = 0.387"
d = 1.25" f = 0.462"
c = 0.350" g = 0.600"
Casting 4-1-2, Specimens Marked "A"
Casting 4-2-1, Specimens Marked "B"
Casting 3-1-2, Specimens Marked "D"
Casting 3-2-3, Specimens Marked "E"
r = 1"
d = 0.375"
Tool Marks Removed with a File
Standard Thread
d = 0.675"
0.75"
0.50"
3"
0.50"
0.75"
5.50"
(d) Small Tension Specimen

FIG. 3.—Castings and Specimens.

(*a*) Castings (*b*) Torsion specimens
(*c*) Location of tension specimens (*d*) Tension specimen

Tests:

Torsional tests were made in a 230,000 in-lb., Olsen, power-driven machine. Strains were measured over a gage length of 5 in. by means of a wire-wound dial instrument on which one division on the dial equals 0.0001 radian per inch. Readings were taken at close intervals, one or two divisions, in the lower range of loading and at intermediate values up to five or ten divisions in the higher ranges of loading. Fracture occurred in the regular helical manner for all specimens tested in torsion. There was no evidence of a buckling failure even with the thinnest cylinders.

When the tension test specimens were being machined it was found that a number of them had blowholes or other defects, which made it necessary to discard them, but otherwise all of the material in all of the tests reported appeared to be reasonably uniform in structure. The tension tests were made in a 10,000-lb. hand-power machine and strains were measured with an attached extensometer, reading directly to 0.0001 in. per inch, which remained on the specimen until rupture.

In addition to the tests reported, a series of torsion and tension tests were made on cylinders from a different heat of iron. A discrepancy appears to exist between the results of the torsion and tension tests of this series. It is thought that further tests will clear up the discrepancy, but since there was not time to make these further tests it seemed best not to include any of the data for that heat of iron in this paper. It seems quite likely, however, that the results will corroborate those given herein as the general trend of the results is the same.

TEST RESULTS AND ANALYSIS OF TEST DATA

The data for the tension tests are given in Table I and for the torsion tests in Table II. Typical stress-strain curves for the tension tests are shown in Fig. 4 and similar twisting moment - angle of twist curves for the specimens tested in torsion are shown in Fig. 5.

TABLE I.—DATA FOR TENSION TESTS.

Specimens with numbers 1 to 8 following the T, as T7, are from the upper portion of the casting; those with numbers 9 to 16 are from the lower portion of the casting. In both upper and lower portions the number relates the location of the tension specimen in the casting to the location of the wall of a cylinder tested in torsion. The smaller numbers are for specimens nearest the inner wall of the casting and the larger the number the farther is the specimen from the inner wall.

Specimen	Corresponding Specimens Tested in Torsion	Diameter, in.	Maximum Load, lb.	Ultimate Tensile Strength, psi.	Maximum Tensile Unit Strain, in. per inch
4-1-2T3	4-1-1G4	0.375	3882	35 100	......
4-1-2T7	4-1-3G3	0.375	3787	34 300	0.00565
4-1-2T12	4-1-4G2	0.374	3645	33 100	0.00500
4-1-2T16		0.373	3878	35 500	0.00660
Average				34 500	0.00575
3-1-2T5	3-1-4G3	0.374	3670	33 300	0.00495
3-1-2T14	3-1-5G4	0.374	3640	33 100	0.00430
Average				33 200	0.00462
4-2-1T3	4-2-3G1	0.374	2395	21 800	0.00260
4-2-1T10	4-2-4G2	0.374	2120	19 300	0.00175
Average				20 500	0.00218
3-2-3T10	3-2-4G7	0.375	1820	16 500	0.00463
3-2-3T12	3-2-5G2	0.375	1820	16 500	0.00420
3-2-3T16		0.374	1830	16 600	0.00370
Average				16 500	0.00418

TABLE II.—DATA FOR TORSION TESTS.

Specimen	Diameter, in.		Wall Thickness, in.	Ratio, Wall Thickness to Outside Radius	Maximum Twisting Moment, in-lb.	Modulus of Rupture, psi.	Maxmium Unit Strain, in. per inch		Modulus of Elasticity, psi.
	Outside	Inside					Shear	Tension	
3-2-4G7	3.150	1.752	0.699	0.443	105 600	19 100	0.0194	0.0097	3 440 000
3-1-5G4	2.599	1.753	0.423	0.325	98 300	36 000	0.0257	0.0129	5 840 000
3-1-4G3	2.364	1.816	0.274	0.232	52 500	31 000	0.0177	0.0089	7 100 000
3-2-5G2	2.210	1.816	0.197	0.178	17 800	15 400	0.0123	0.0062	5 600 000
4-1-1G4	4.002	3.252	0.375	0.187	236 000	33 300	0.0300	0.0150	6 530 000
4-1-3G3	3.844	3.251	0.296	0.154	184 800	33 900	0.0313	0.0157	6 250 000
4-1-4G2	3.647	3.251	0.195	0.107	105 500	30 100	0.0192	0.0096	6 090 000
4-2-4G2	3.607	3.253	0.177	0.098	48 200	15 500	0.0133	0.0067	3 180 000
4-2-3G1	3.450	3.252	0.099	0.057	25 500	15 100	0.0086	0.0043	3 160 000

The tension tests were made primarily to obtain the ultimate tensile strength and the ultimate tensile strain. While typical curves are plotted in order to show the general stress-strain charac-

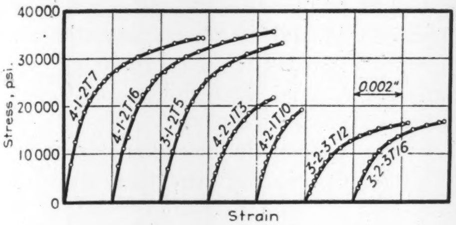


FIG. 4.—Typical Stress-Strain Curves in Tension.

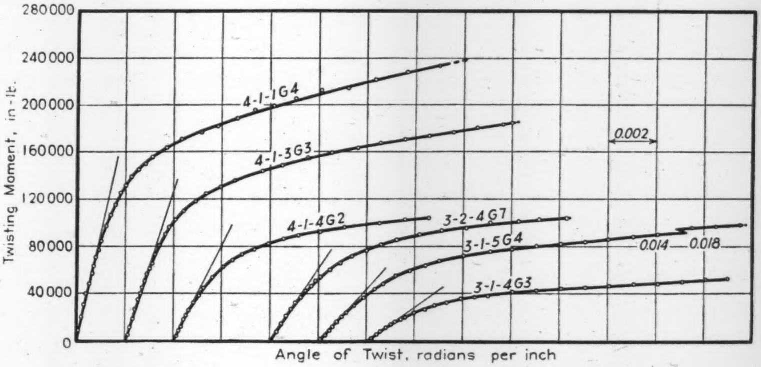


FIG. 5.—Typical Twisting Moment - Angle of Twist Curves.

teristics of the cast irons, it is not considered that the curves are sufficiently reliable to be used for the determination of the modulus of elasticity. It will be observed that the ultimate tensile strain for the 4½-in. casting of heat No. 2 is only about one-half the ultimate tensile strain of the 3½-in. casting from the same heat. The 3½-in. casting was poured after the 4½-in. casting and there may have been some change in the iron in the cupola between the two pourings.

A comparison is made in Fig. 6 of the ratio of modulus of rupture in torsion to tensile strength in direct tension (strength ratio) and of the ratio of maximum tensile strain in torsion (tensile strain is assumed to be one-half the shearing strain) to maximum tensile strain in tension (strain ratio) with the ratio of wall thickness to the outside radius. Both the strength ratio and the strain ratio are based on the average values of tensile strength and strain (see Table I) for each heat and size of casting. In Fig. 6 are plotted also the values obtained for these quantities from similar tests made and reported in 1938 and 1939.

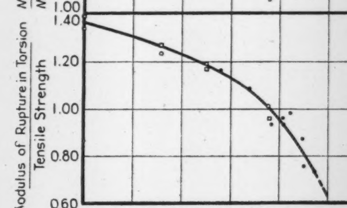


FIG. 6.—Curves Showing Relationship in Torsion Between Modulus of Rupture and t/r and Between Maximum Tensile Strain and t/r.

Since the tests herein reported were made in response to questions concerning the validity of the application of previous tests on cylinders only 1 in. in diameter to larger cylinders and also to smaller ratios of t/r than it was possible to obtain with the smaller cylinders, the tests reported here cover a relatively smaller range than the earlier tests. However where they overlap the earlier tests there is an excellent agreement, at least so far as the strength ratios are concerned. The strain ratios, taken as a whole, do not give as consistent results as the strength ratios, but they do show that for a given iron there is a definite decrease in the maximum unit strain obtained as the ratio t/r decreases. However, after t/r has decreased to about 0.2 there seems to be no further decrease in the strain ratio. Some of the scatter in the strain ratios for the lower values of t/r is due to the low values of the maximum strains found for tension specimens 4–2–1T3 and 4–2–1T10, because it is on the maximum strains of these specimens that two of the high values of strain ratios are based.

In considering the closer agreement between the strength ratios as compared with the strain ratios, it should be borne in mind that such a variation may be expected. Because of the relatively flat portion of the upper part of the stress-strain diagrams, a failure due to the presence of some minor defect may cause the ultimate strength to be reduced only about 10 per cent while the maximum strain will be reduced as much as 35 per cent.

CONCLUSIONS

1. The tensile strengths of the specimens from the different castings are quite uniform within each heat (maximum variation from average is about 4 per cent) but differ between heats; the maximum tensile deformations are reasonably uniform within each heat except in one case.

2. There is no evidence of any buckling failure in any of the thin cylinders.

3. The results of the tests on hollow cylinders $3\frac{1}{2}$ and $4\frac{1}{2}$ in. in diameter are in agreement with tests previously reported for solid and hollow cylinders having diameters of 1 in.

4. The ratio of the modulus of rupture in torsion to the ultimate tensile strength in direct tension (strength ratio) is independent of the external diameter of the cylinder; The variation of this ratio for a given quality of iron depends only upon the ratio of the wall thickness to the outside radius.

5. For all tests reported, the ratio of the modulus of rupture in torsion to the ultimate tensile strength in direct tension (strength ratio) varies in a smooth curve from 1.36 for a solid cylinder to 0.73 for a thin-walled cylinder having a ratio of wall thickness to outside radius of 0.057. This curve, when extrapolated, gives a strength ratio of about 0.63 for zero t/r.

6. The ratio of the maximum tensile deformation developed in torsion to the maximum tensile deformation developed in tension (strain ratio) decreases as the ratio of wall thickness to outside radius decreases until a t/r value of about 0.2 is reached after which there is no further definite change.

Acknowledgments:

This investigation was carried out as a part of the work of the Engineering Experiment Station of the University of Illinois. The castings were made and machined in the Machine Laboratories of the University of Illinois, the immediate supervision of the work being given by J. F. Wooddell, Associate in Mechanical Engineering, and C. E. Schubert, Assistant Professor of Mechanical Engineering. The chemical analyses and the photomicrographs were obtained through the courtesy of J. T. MacKenzie of the American Cast Iron Pipe Co.

DISCUSSION

Mr. H. F. Moore.[1]—In the results of torsion tests of cast iron quoted in this paper there is an indication that the fracture of cast iron follows the maximum strain theory rather than the maximum stress theory.

In a torsion specimen the maximum shearing stress is either on a longitudinal section or on a cross-section (horizontal or vertical). On one of the sections making an angle of 45 deg. with the maximum shearing stress there is a tensile stress equal in magnitude to the maximum shearing stress. On the other 45-deg. section there is a compressive stress of the same magnitude.

The shearing strength of cast iron is greater than its tensile strength and torsion specimens of cast iron fail along the "tensile" 45-deg. section.

Now if the maximum stress theory holds, the stress at fracture for cast iron should be the same for a torsion test of a hollow specimen as for a tension test. If the maximum strain theory holds, the compressive stress on one 45-deg. plane of the torsion specimen would lower the resistance to fracture on the other 45-deg. plane (Poisson's ratio effect). The latter seems to be the case.

This result is in harmony with results obtained a good many years ago by two Japanese experimenters Matsumura and Hamabe.[2] Gough's fatigue tests[3] of cast iron under combined stress yield results falling between the two theories.

Mr. J. M. Lessells.[4]—I should like to ask the authors whether they believe that the differences in the materials, as evidenced by the photomicrographs, would influence the results.

Messrs. J. O. Draffin[5], W. L. Collins[5] and C. H. Casberg[6] (*Authors' closure by letter*).—Reference is made in the paper to tests on a third heat of iron, the data for which were not reported. In this third heat, similar to heat No. 1 except that heat No. 3 contained 1.05 per cent nickel and 0.44 per cent molybdenum, the tensile strengths of specimens cut from the two No. 5 cylinders, 3½ in. and 4½ in., were much lower than was expected. From a study of the pouring procedure for this heat and the general appearance of the iron, as well as the strength properties, it became clear that the iron in the No. 5 cylinders was not like that in the cylinders tested in torsion and consequently the data from these tension test specimens were discarded. In order to overcome this lack of knowledge of the tensile strength of heat No. 3, ⅜-in. specimens were cut from the rectangular ends of two of the cylinders tested in torsion, 3–3–2G3 and 4–3–3G4, and tested. Because the rectangular ends of the cylinders were short, the gage length of the tension test specimens was only 1 in. and consequently no strain measurements were made. The tensile strengths obtained from these specimens are believed to represent the average tensile strengths of the cylinders tested in torsion. The properties in tension and torsion and the ratios of the modulus of rupture to the tensile strength are given in the accompanying Table I.

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[1] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] T. Matsumura and G. Hamabe, "Tests on the Combined Bending and Torsional Strength of Cast Iron," *Memoirs*, College of Engineering, Kyoto Imperial University, Vol. 1, No. 2, February, 1915, 85 pp.

[3] H. J. Gough and H. V. Pollard, "The Strength of Materials Under Combined Alternating Stresses," (see discussion), *Proceedings*, Inst. Mechanical Engrs. (British), Vol. 131, pp. 3-103 (1935).

[4] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[5] Professor, and Assistant Professor, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[6] Professor of Mechanical Engineering, University of Illinois, Urbana, Ill.

Comparison of these ratios with those shown in Fig. 6 shows that the strength curve should be somewhat flatter at the smaller ratios of *t*/*r* than the one shown. It would appear from all the data which have been presented that the strength ratios will remain approximately constant for *t*/*r* ratios of less than about 0.20. It is probable that for average irons of uniform quality few strength ratios will fall below 0.85, which is the value reached when *t*/*r* is about 0.2.

TABLE I.—PROPERTIES OF CAST IRON FROM HEAT NO. 3.

Specimen	Ratio of Wall Thickness to Outside Radius	Torsion				Tensile Strength, psi.	Ratio of Modulus of Rupture in Torsion to Tensile Strength
		Modulus of Rupture, psi.	Maximum Strain, in. per inch		Modulus of Elasticity in Shear, psi.		
			Shear	Tension			
3-3 3G7	0.443	36 500	0.0164	0.0082	5 600 000		1.010
3-3-4G4	0.325	32 000	0.0168	0.0084	5 090 000	(Avg. of 4 tests, 36,100)[a]	0.888
3 3 2G3	0.238	34 900	0.0161	0.0081	6 000 000		0.966
3-3 1G2	0.186	33 800	0.0101	0.0051	6 000 000		0.965
4-3 3G4	0.185	31 700	0.0162	0.0081	5 160 000	(Avg of 3 tests, 32,200)[a]	0.985
4-3 4G3	0.156	29 200	0.0145	0.0073	4 500 000		0.908
4-3-2G1	0.059	31 500	0.0135	0.0068	5 200 000		0.978
4-3-1G1	0.054	29 600	0.0095	0.0048	5 870 000		0.920

[a] Maximum variation, 5.8 per cent above and 5.6 per cent below.

The suggestion of Mr. Moore that the results of these tests constitute evidence supporting the maximum strain theory is interesting. According to this theory, and assuming Poisson's ratio as 0.25 and the modulus of elasticity constant and the same in tension and compression, the reduction in tensile strength for the thin cylinders would be about 20 per cent, while the tests show a decrease of about 15 per cent. Apparently this affords some confirmation of the maximum strain theory. However, when strains are compared the evidence is not as convincing. Taking an average of many tension tests the average maximum tensile strain for cast iron is roughly 0.006 in. per inch and, according to the maximum strain theory, failure should occur when the thin cylinders are strained in tension to this amount. However, most of the cylinders tested in torsion developed tensile strains somewhat above 0.006 in. per inch. But it should be remembered that there is little information on the value of Poisson's ratio for cast iron and but little real knowledge of the action of cast iron under combined stress. Furthermore, for reasons which it would take too long to discuss, it is the opinion of the authors that some of the larger strain ratios shown in Fig. 6 for small values of *t*/*r* are not as reliable as the smaller strain ratios. Consequently it may be concluded that the strength values tend to confirm the maximum strain theory while the strain values do not do so, although they do not definitely contradict it.

STRESS-STRAIN RELATIONS FOR MALLEABLE CAST IRON IN TENSION WITH SPECIAL ATTENTION TO YIELD POINT DETERMINATION[1]

By R. D. Landon[2]

Synopsis

The physical behavior of 141 tension specimens of malleable cast iron covering a wide range of carbon content is recorded and, based on the results, changes in the standard testing procedure are suggested for consideration. Results indicate that "yield point" stress might better be expressed as a percentage of the ultimate stress rather than as an arbitrarily fixed value. An elongation in 2 in. of 0.02 in. is recommended for the divider method of measuring deformation at "yield point" and a stress of 63 per cent of ultimate for minimum "yield point" stress is suggested. If an elongation in 2 in. of 0.01 in. is to be used, the suggested minimum "yield point" stress is 60 per cent of ultimate and a more accurate means of measurement should be employed. In order to be consistent with the Society's definition of yield point, a new term, "proof stress," is suggested for the stress to be equalled or exceeded at a definite, measured elongation. Confirmation of negligible reduction in the strength of machined castings is mentioned.

This study of certain physical properties of malleable cast iron, undertaken at the invitation of the Society's Committee A-7 on Malleable Iron Castings, was for the specific purpose of obtaining additional data on "yield point" stress. At the committee's request, twelve manufacturers furnished a total of 141 specimens. Included in this number were some specimens whose carbon content was outside the range on which the present testing specifications are based.

The specimens apparently from each foundry were arranged in one group and labeled. Such groupings were made only to avoid unnecessary adjustment of the jaws of the testing machine. All identifying marks of the manufacturer were removed and no significance was attached to the groupings.

Three groups, A, C, and D, which appeared to be the most accurately cast, were tested first.

One specimen from each of the remaining 9 groups was turned down to $\frac{1}{2}$-in. diameter in the gage length and about $\frac{11}{16}$ in. in the grip sections. Each specimen so prepared has been designated with a "t" as B-1t. The remaining specimens in each group were divided equally into two sets, one of which was turned down in the grip sections only by clamping the gage length in a self-centering lathe chuck. This procedure reduced eccentricity to a minimum and made possible more accurate measurements with a strain gage which was attached to specimens of these sets. All specimens of such sets have been designated with a "m" as B-4m. The other set of each group, if reasonably round in the grip sections and also straight, was tested as cast. Such specimens have been designated with a "p" as B-9p and were tested with an extensometer (one division corresponding to 0.0005 in. elongation) attached. In several groups every specimen was machined in the grip sections in order to reduce eccentricity or bend. As the strain gage indicates much smaller units than the extensometer, it was used on the machined sets in order to determine the stress-strain relations on both sides of the proportional limit. From these data corrected stress-strain diagrams were constructed and moduli of elasticity computed. (The maximum elongation obtainable with the strain gage was 0.02 in.)

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[1] Condensation of a report submitted to Committee A-7 on Malleable Iron Castings.

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DISCUSSION OF RESULTS

As indicated by the stress-strain diagrams (Fig. 1) all specimens had essentially the same characteristics. The proportional limit was reached at a stress of approximately 20,000 psi. and a strain of approximately 0.001 in. per inch but values as low as 16,000 and as high as 27,000 psi. were observed. None of the specimens exhibited a definite "yield point." *There was no drop of the beam of the testing machine and no dip in the stress-strain diagrams.*

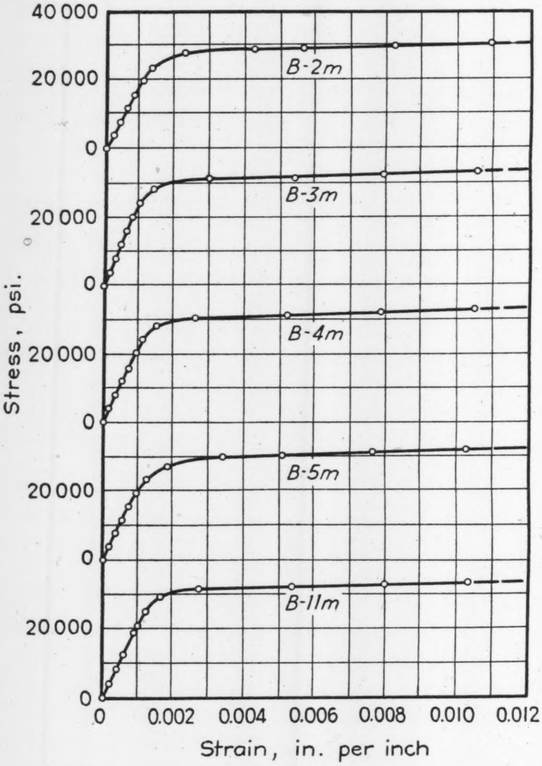

FIG. 1.—Stress-Strain Curves for Group B.

Above the proportional limit the stress-strain diagrams flattened out in a smooth curve and then continued in practically a straight line having a very flat slope.

TABLE I.—MODULUS OF ELASTICITY.

Specimen	Modulus of Elasticity, psi.	Specimen	Modulus of Elasticity, psi.
No. B-1t	21 000 000	No. I-1t	24 000 000
No. B-2m	20 000 000	No. I-2m	25 000 000
No. B-3m	26 000 000	No. I-3m	18 000 000
No. B-4m	22 000 000	No. I-4m	26 000 000
No. B-5m	22 000 000	No. I-5m	24 000 000
No. B-11m	21 000 000	No. I-6m	27 000 000
No. E-1t	24 000 000	No. J-1t	33 000 000
No. E-2m	22 000 000	No. J-2m	19 000 000
No. E-3m	23 000 000	No. J-3m	20 000 000
No. E-4m	23 000 000	No. J-4m	29 000 000
No. E-5m	21 000 000	No. J-5m	25 000 000
No. E-11m	22 000 000		
		No. K-1t	22 000 000
No. F-1t	26 000 000	No. K-2m	23 000 000
No. F-2m	23 000 000	No. K-3m	16 000 000
No. F-3m	23 000 000	No. K-4m	20 000 000
No. F-4m	23 000 000	No. K-6m	23 000 000
No. F-5m	27 000 000		
No. F-11m	33 000 000	No. L-1t	22 000 000
		No. L-2m	18 000 000
No. G-1t	22 000 000	No. L-3m	19 000 000
No. G-2m	22 000 000	No. L-4m	17 000 000
No. G-3m	21 000 000	No. L-5m	18 000 000
No. G-4m	21 000 000	No. L-6m	20 000 000
No. G-5m	20 000 000		
No. G-11m	19 000 000		
No. H-1t	21 000 000		
No. H-2m	22 000 000		
No. H-3m	25 000 000		
No. H-4m	21 000 000		
No. H-5m	22 000 000		
No. H-11m	23 000 000		

Average for all 52 specimens = 22,500,000 psi.
Average for 9 turned specimens = 24,000,000 psi.
Average for turned specimens excluding J-1t = 23,000,000 psi.

TABLE II.—STRESS-STRAIN RELATIONS—GROUP B.

All specimens in Group B not round in grip section.

	Elongation in 2 in., in.	B-1t	B-2m	B-3m	B-4m	B-5m	B-6m	B-7m	B-8m	B-9m	B-10m	B-11m	B-12m
Stress, psi	0.002	21 000	20 000	24 600	22 000	20 200	24 600	23 800	22 200	20 900	23 600	21 100	.....
	0.005	32 400	28 300	31 000	30 500	29 000	.....	30 200	29 100	28 500	29 200	31 300	.....
	0.010	34 600	29 300	32 000	31 400	30 300	32 200	31 200	30 500	30 500	30 400	32 000	.....
	0.020	36 800	30 600	33 200	33 000	32 000	33 100	32 000	31 600	32 000	31 500	33 400	.....
	0.050	.....	.....	.....	.....	.....	37 000	36 800	36 400	36 400	35 700	.....	.....
Ultimate Stress, psi		55 500	49 600	53 100	53 000	51 800	[a]	51 500	47 900	48 400	49 000	50 300	52 700
Elongation, per cent		[b]	16.5	18.0	20.0	18.0	10.5	22.5	11.0	12.5	16.0	12.0	17.0

[a] Slight surface blow hole—failure through blow hole.
[b] Failed outside gage length but in net section.

The modulus of elasticity (Table I) varied from 16,000,000 to 33,000,000 psi. with an average value for 52 specimens of 22,500,000 psi. The great discrepancy in these values results in part at least from the fact that some of the specimens were bent. The average value, however, should be reasonably correct. The only specimens which were well suited for this determination were those completely turned (B-1t, E-1t, etc.). The range on these 9 turned specimens was from 21,000,000 to 33,000,000 psi. with an average value of 24,000,000 psi. Excluding the one specimen for which the modulus was 33,000,000 psi. (a value abnormally high) the range was from 21,000,000 to 26,000,000 with an average of 23,000,000 psi., a value which checks very closely with the above average for all specimens. Incidentally this close agreement between the average value for turned specimens and those on which the net section was undisturbed substantiates further the contention that machining malleable castings has no appreciable effect on stress and strain within the elastic range. A further comparison will be made later when "yield point" stress, ultimate stress, and percentage elongation are discussed.

In Table II the stresses developed at elongations of 0.002, 0.005, 0.010, 0.020 and 0.050 in. in 2 in., ultimate stress and percentage elongation are recorded for group B. The stress at an elongation of 0.002 in. in 2 in. was observed because the proportional limit was reached at approximately those values. If malleable iron had a *definite* yield point such as mild steel exhibits, it

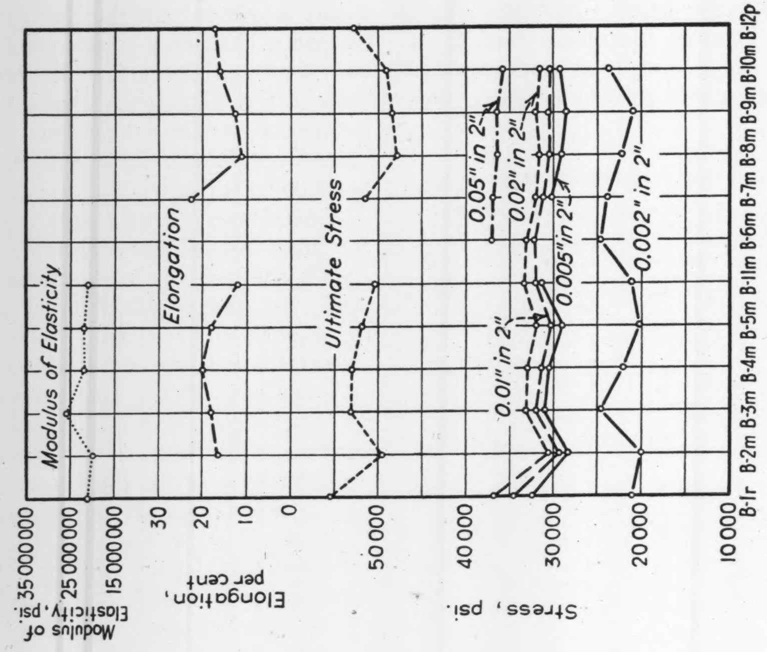

FIG. 3.—Stress-Deformation Curves for Group B.

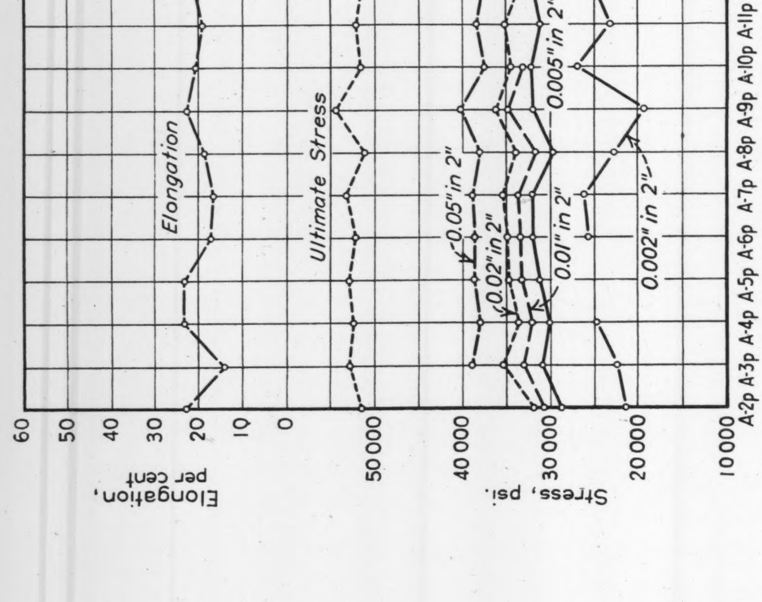

FIG. 2.—Stress-Deformation Curves for Group A.

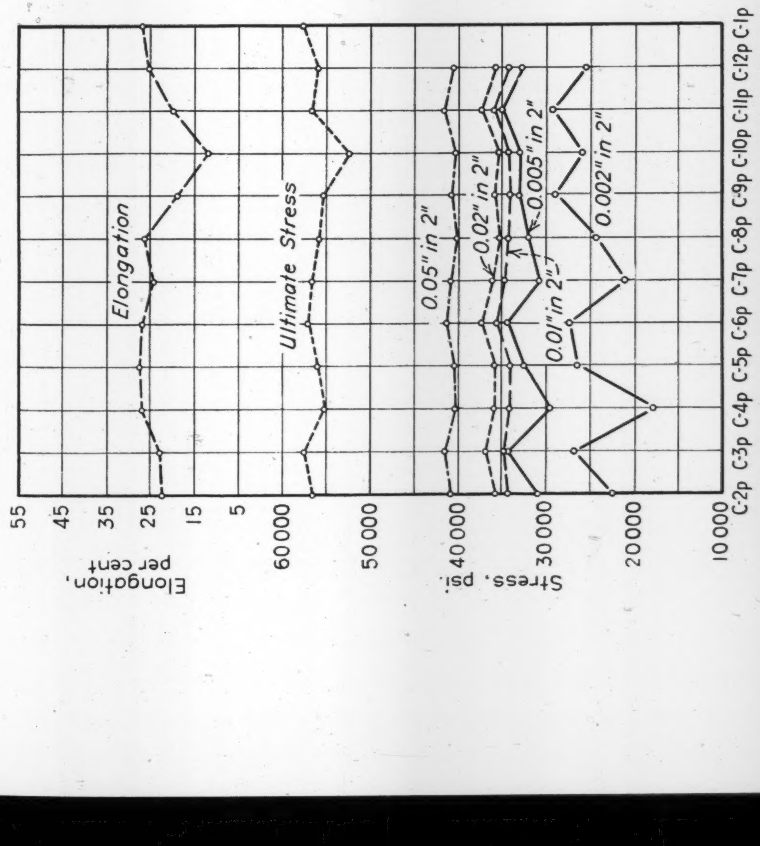

FIG. 4.—Stress-Deformation Curves for Group C.

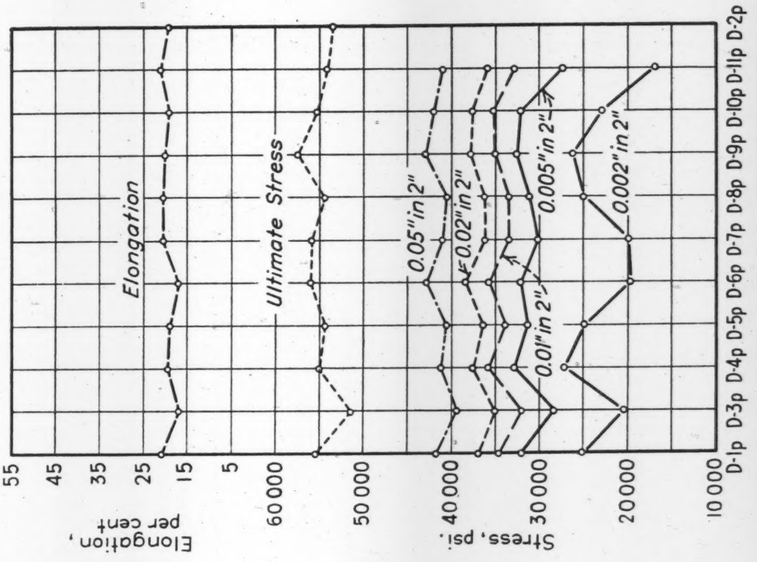

FIG. 5.—Stress-Deformation Curves for Group D.

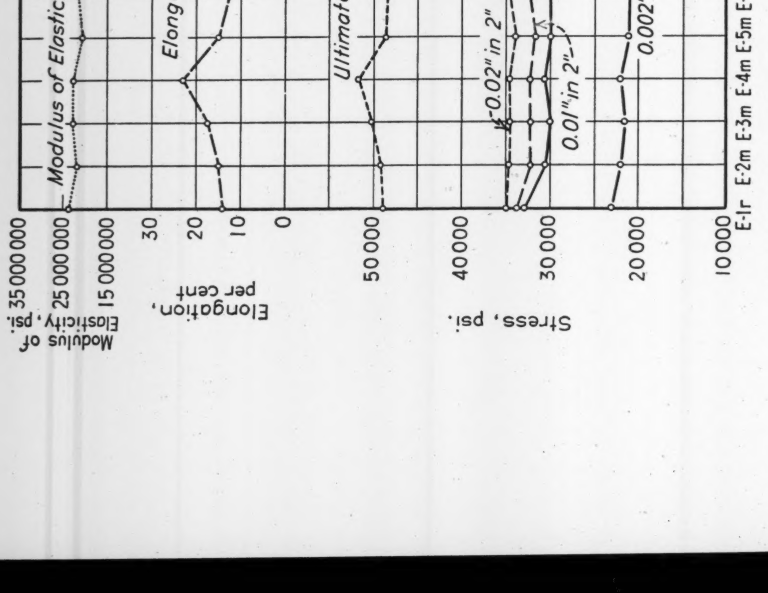

Fig. 6.—Stress-Deformation Curves for Group E.

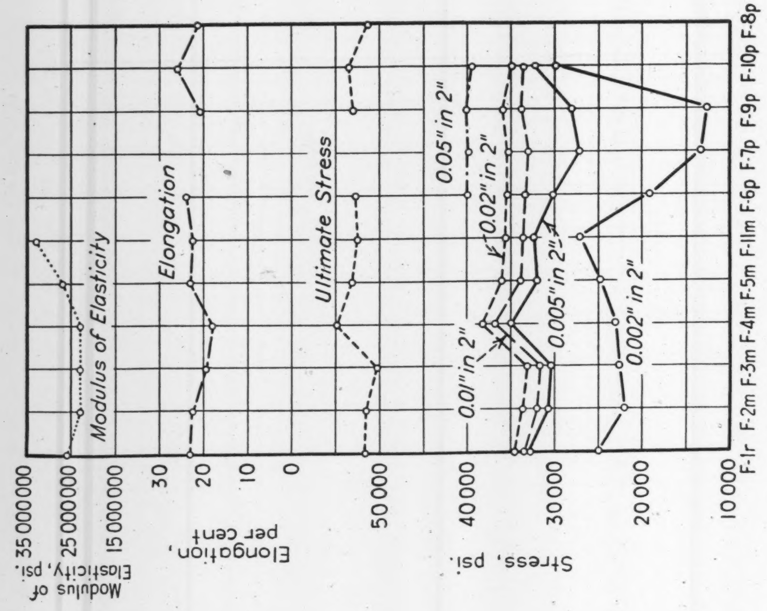

Fig. 7.—Stress-Deformation Curves for Group F.

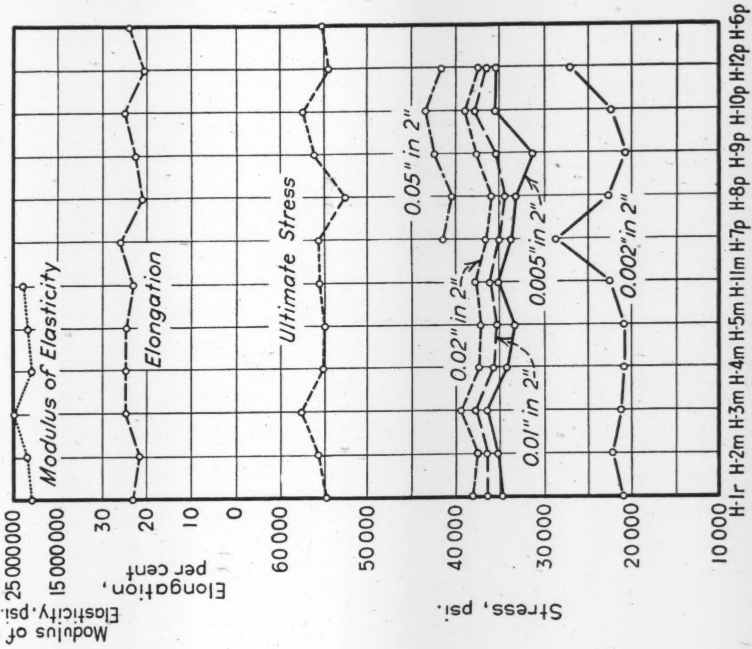

FIG. 9.—Stress-Deformation Curves for Group H.

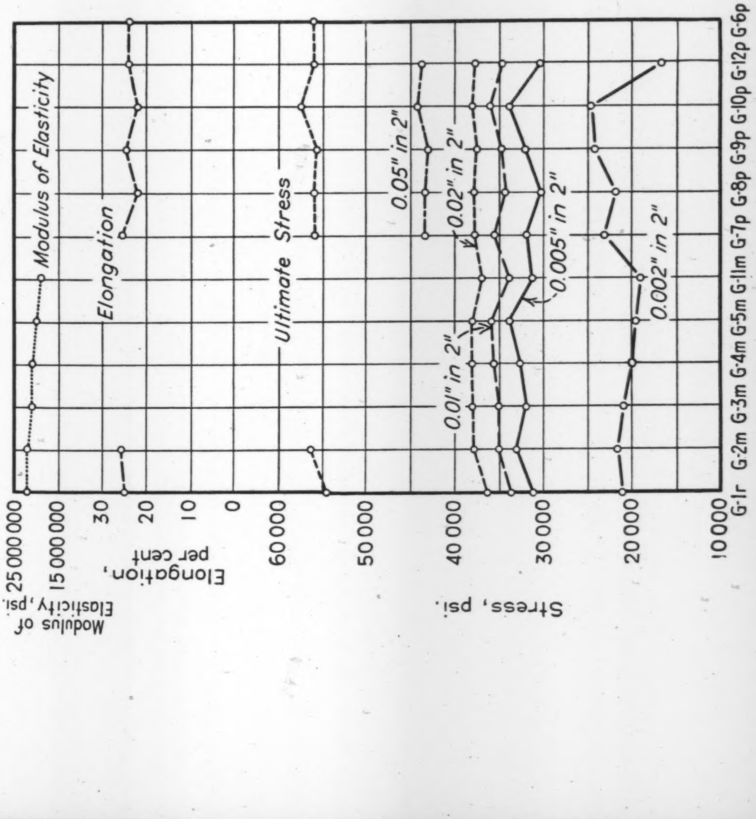

FIG. 8.—Stress-Deformation Curves for Group G.

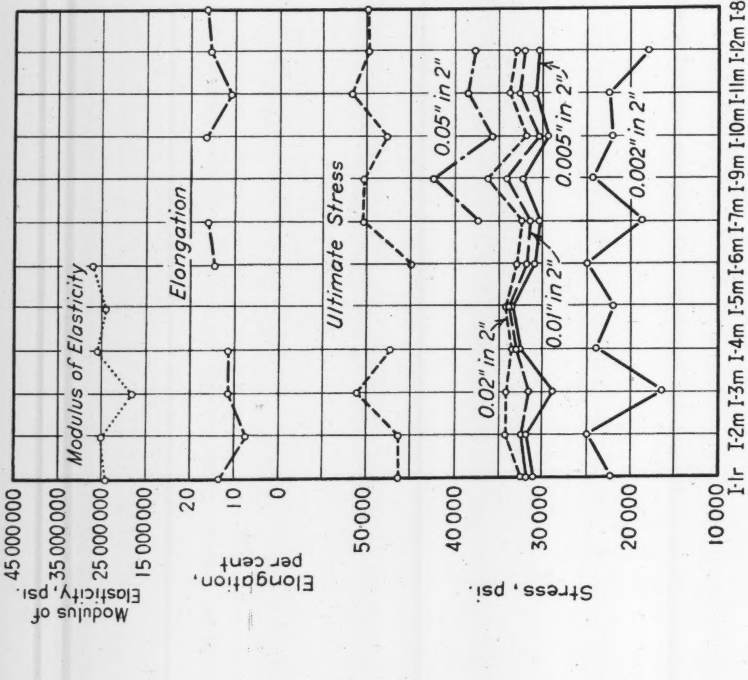

FIG. 10.—Stress-Deformation Curves for Group I.

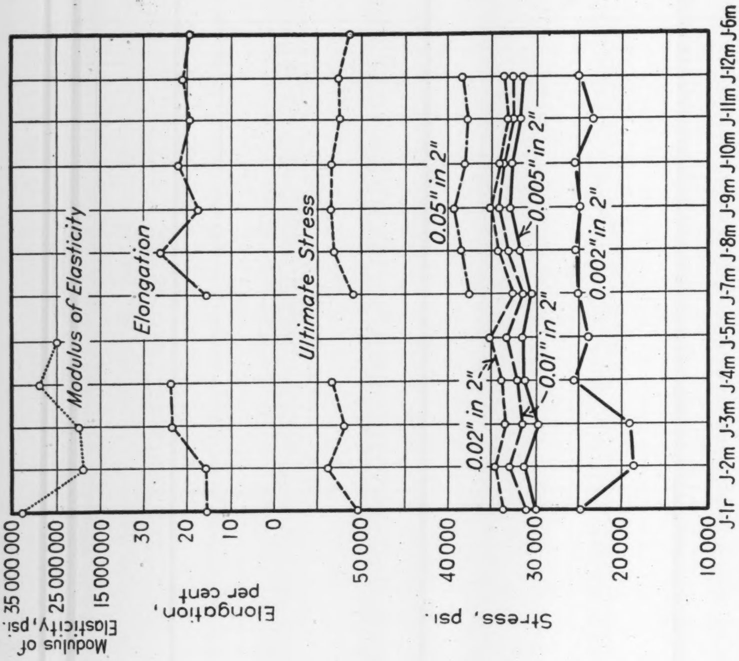

FIG. 11.—Stress-Deformation Curves for Group J.

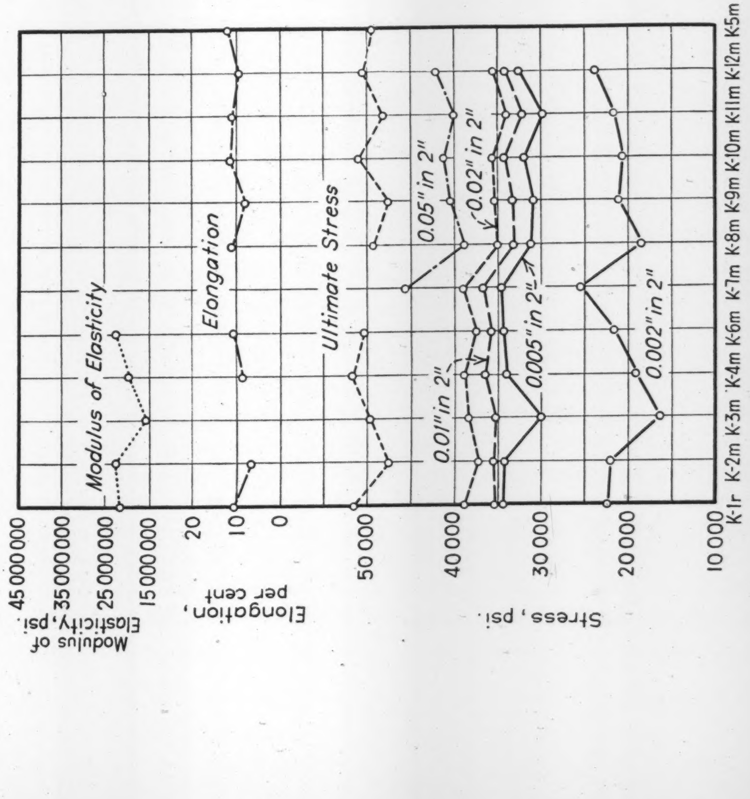

FIG. 12.—Stress-Deformation Curves for Group K.

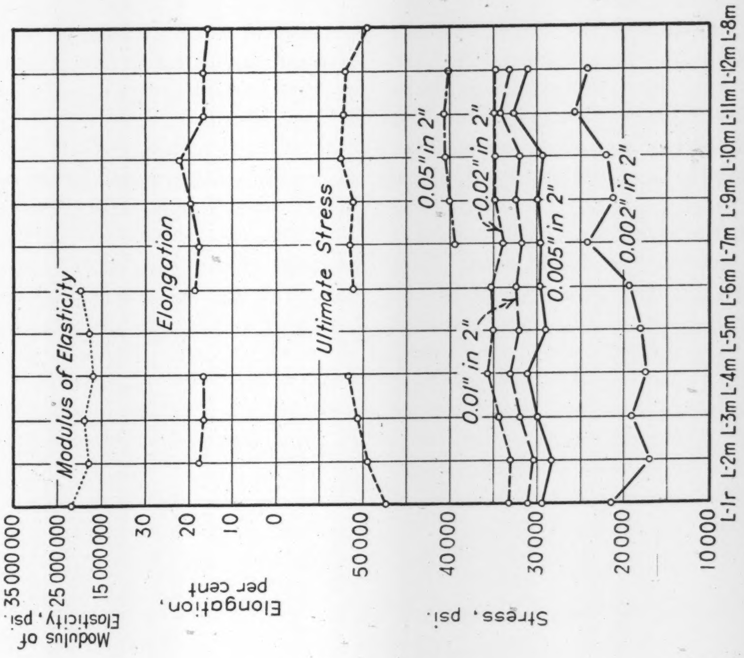

FIG. 13.—Stress-Deformation Curves for Group L.

would occur at practically that same location. The stress at an elongation of 0.005 in. in 2 in. was observed because that point on the stress-strain diagram is perhaps the first which might be used without reference to other stress-strain measurements as an indicator of yield. In other words, this point is in the flat portion of the stress-strain diagram and it might serve as the "yield point" just as properly as the stress at 0.010 or 0.020 in. in 2 in. Obviously a rather accurate instrument for measurement of 0.005 in. in 2 in. would be required.

Figures 2 to 13 show the correlation of results for all the specimens. While no calculations have been made to establish definite relations between most of the values shown, there appears to be a rather definite relation between the stress developed at 0.010 and 0.020-in. elongation. The stresses at other elongations seem to be more nearly independent of each other. Some relation between ultimate stress and percentage elongation may exist but it is not very well defined. Further work on correlation might reveal interesting and useful information.

TABLE III.—MINIMUM, MAXIMUM, MEAN AND AVERAGE VALUES—GROUP B.

	Elongation in 2 in., in.	Minimum	Maximum	Mean	Average	Maximum Deviation from Mean, per cent	Maximum Deviation from Average, per cent	
							Plus	Minus
Stress, psi......	0.002....	20 000	24 600	22 300	22 181	10.3	10.8	9.9
	0.005....	28 300	32 400	30 350	29 950	6.8	8.0	5.7
	0.010....	29 300	34 600	31 950	31 309	8.3	10.5	6.4
	0.020....	30 600	36 800	33 700	32 655	9.2	12.5	6.4
	0.050....	35 700	37 000	36 350	36 460	1.8	1.4	2.2
Ultimate Stress, psi..........		47 900	55 500	51 700	51 164	7.4	8.4	6.4
Elongation, per cent.........		10.5	22.5	16.5	16.35			

In Table III the minimum, maximum, mean and average values of stress for group B at the several elongations are shown.

In addition maximum percentage deviation from mean and average values has been tabulated for comparison. Examination of the deviation calculations indicates that the maximum percentage deviation in stress from either average or mean values at 0.002 in. in 2 in. is approximately three times the maximum percentage deviation at 0.005 in. in 2 in. As the maximum deviations in stress at 0.005, 0.010, and 0.020 in. in 2 in. are relatively low and as the average of these maximums agrees very closely with the average for ultimate strength maximum deviation, there is little theoretical choice between these three values of elongation and their respective stresses as indicators of yield. A minimum value of stress to be attained at any of the three elongations could be selected and defined as the stress at "yield point." For commercial testing procedure, however, it would seem wise to select the elongation which could be measured most accurately with ease and a minimum of equipment. To satisfy this suggestion, the author favors an elongation of 0.020 in. in 2 in. if the divider method is to be used.

Consideration should be given to values of stress produced at 0.005, 0.010 and 0.020-in. elongation. Summarized results for 129 specimens are tabulated below:

Elongation in 2 in., in.	Minimum Stress, psi.	Maximum Stress, psi.	Average Stress, psi.
0.005......	27 200	36 500	31 400
0.010......	29 300	37 800	33 500
0.020......	30 600	39 400	35 300

The average stress at 0.010-in. elongation is 2100 psi. more than at 0.005 in. and the average at 0.020-in. elongation is 1800 psi. more than at 0.010 in. In other words, the average stress at "yield point" could be 31,400, 33,500 or 35,300 psi. depending on the elongation selected and the differences would be 2100 and 1800 psi., respectively.

Comparison of averages for the 9 turned specimens only and the total of 129 specimens is as follows:

Elongation in 2 in., in.	Average Stress, psi. Turned Specimens	Average Stress, psi. Total Specimens
0.005...........	32 100	31 400
0.010...........	33 500	33 500
0.020...........	35 400	35 300

In this comparison also, there is very little difference between the behavior of turned specimens and those as cast.

TABLE IV.—PERCENTAGE OF ULTIMATE STRESS AT SEVERAL ELONGATIONS—GROUP B.

Elongation in 2 in., in.	B-1t	B-2m	B-3m	B-4m	B-5m	B-6m	B-7m	B-8m	B-9m	B-10m	B-11m	B-12m
0.002....	37.8	40.3	46.3	41.5	39.0	....	46.2	46.4	43.2	48.1	42.0	...
0.005....	58.4	57.0	58.4	57.5	56.0	....	58.6	60.8	58.9	59.6	62.2	...
0.010....	62.3	59.0	60.3	59.3	58.5	....	60.6	63.7	63.0	62.0	63.6	...
0.020....	66.3	61.6	62.5	62.3	61.8	....	62.1	66.0	66.1	64.3	66.4	...
0.050....	....	....	....	....	....	....	71.4	76.0	75.2	72.8	....	...

Results on ultimate stress and percentage elongation for group B are given in Table II and summarized in Table III. For 131 specimens the average ultimate stress was 52,400 psi. with a range from 45,000 to 57,700 psi. The average ultimate stress for the 9 turned specimens was 51,200 psi. or 1200 psi. less than the average for all specimens. The average elongation for 128 specimens was 18.8 per cent with a range from 6.5 to 27.5 per cent. For the 9 turned specimens the average was 17.8 per cent or 1.0 per cent less than the average for all specimens. This comparison indicates that removal of the "skin" reduced slightly the ultimate stress and the percentage elongation but too general conclusions should not be drawn from a limited number of tests.

As the proportional limit was reached when the elongation in 2 in. was approximately 0.002 in., calculations were made to obtain ratios between stress at 0.002 in. elongation and stress at both 0.010 and 0.020 in., but no consistent results could be obtained. Then an attempt was made to express the stresses at 0.005, 0.010 and 0.020-in. elongation as percentages of the ultimate stress. The results for group B are shown in Table IV. The ultimate stress was chosen as a basis of comparison because the independence of elongation measurement should make it the most accurate value determined. While the ratios varied considerably, the author believes that a minimum ratio of stress at either 0.005, 0.010 or 0.020-in. elongation to the ultimate stress might prove a more satisfactory basis for "yield point" determination than the present specification of a *definite value* for *all* specimens. If the specifications called for *first*, ultimate stress of at least 50,000 psi. and *second*, "yield point" stress of at least X per cent of the ultimate they would be more in keeping with the properties of the specimens tested. The results show that while 79.4 per cent of all specimens developed an ultimate stress of at least 50,000 psi., only 67.2 per cent developed a "yield point" stress of 32,500 psi. The results indicate further that while all but 8 specimens developing a "yield point" stress of 32,500 psi. failed to develop at least 50,000 psi. at the ultimate, 24 specimens developing an ultimate of 50,000 psi. or more failed to show at least 32,500 psi. at "yield point." In other words, if the specimens tested were representative, the present specifications on "yield point" stress are considerably more difficult to meet than the specifications on ultimate stress. It is repeated here that these tests were conducted on material including some metal known to be of higher carbon than would be used if the current specifications were to be met.

Careful examination of the results obtained in these tests indicates that a suggested value of the minimum "*X* per cent" referred to above in the specifications for "yield point" would be:

55 per cent for an elongation of 0.005 in. in 2 in.
60 per cent for an elongation of 0.010 in. in 2 in.
63 per cent for an elongation of 0.020 in. in 2 in.

As the allowable minimum for ultimate stress would be 50,000 psi., the following minimum allowable stresses and corresponding elongations would automatically result:

27 500 psi. at 0.005 in. in 2 in.
30 000 psi. at 0.010 in. in 2 in.
31 500 psi. at 0.020 in. in 2 in.

When these suggested minimum values are checked against all specimens which developed an ultimate stress of at least 50,000 psi., 8 would fail to pass the minimum allowable stress at 0.005 in. elongation, 3 would fail at 0.010 in. and 5 would fail at 0.020 in. elongation. Cross-checking indicates that none of the 8 failing to pass the stress requirement at 0.005 in. elongation also failed the 0.010 or 0.020 in. elongation requirements. On the other hand, however, all 3 failing to qualify for the 60 per cent of ultimate at 0.010 in. elongation failed also to meet the 63 per cent of ultimate at 0.020 in. elongation. Because of these facts consideration of stress at elongation of 0.005 in. in 2 in. as a possible "yield point" probably should be dropped.

It has undoubtedly been noticed that the term *yield point* has appeared in quotations a number of times. This distinction was made advisedly because of the following statement appearing in the A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8 – 36).[3]

> "*Yield Point.*—The stress in a material at which there occurs a marked increase in strain without an increase in stress.
>
> It should be noted that only materials that exhibit this unique phenomenon of yielding have a yield point. The term yield point should not be used in connection with material whose stress-strain curve in the region of yield is a smooth curve of gradual curvature."

With this quotation as a definition and with the included stress-strain diagrams as evidence, the fact is established that malleable iron has no true yield point. As an alternative, the term *proof stress* is offered. Then on the basis of the results of these tests the specifications for malleable cast iron might read:

Tensile Strength, min., psi.	50,000
Proof Stress, min., psi. . .	60 per cent of tensile strength at 0.010 in. in 2 in. or 63 per cent of tensile strength at 0.020 in. in 2 in.

SUMMARY

Turned specimens had practically the same modulus of elasticity and stress at elongations of 0.005, 0.010 and 0.020 in. in 2 in. as did specimens "as cast" but the former had slightly lower values of ultimate stress and percentage elongation.

There was no drop of the beam of the testing machine during any test and reference to such occurrence should be deleted from the A.S.T.M. specifications.

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[3] 1939 Book of A.S.T.M. Standards, Part I, p. 761.

Best values of elongation for determining "yield point" appear to be either 0.010 or 0.020 in. in 2 in. If the smaller value is used, an instrument of greater accuracy than dividers and scale should be used for measurement.

Malleable iron has no true yield point and in order to conform to A.S.T.M. definitions the term "yield point" should not be used. A suggested term, both correct and descriptive, is *proof stress.*

The characteristics of malleable iron as determined in this series of tests indicate that the proof stress might better be specified as a percentage of the ultimate stress rather than as an arbitrarily fixed value.

Many specimens were eccentric or out of round in the grip sections owing to the presence of metal from sprues and risers. In some cases large identifying marks on the grip sections precluded pulling without introduction of eccentric forces. A considerable number of specimens failed to have a straight axis of generation (that is, they were bent). Naturally all of these physical defects have their effect on the accuracy of results and penalize the material under test. More explicit specifications for molding, casting and inspection should pave the way toward improved tension test specimens.

Further consideration of the data and results should lead to additional conclusions and further tests should be conducted by other investigators on specimens from the same and other sources.

DISCUSSION

Mr. H. F. Moore.[1]—I agree fully with the statement in Mr. Landon's conclusions. In order to conform to A.S.T.M. definitions[2] the term "yield point" should not be used.

Mr. Landon suggests that the term "proof stress" be used. There are two objections to the use of this term. First, although the term "proof stress" is not included in the A.S.T.M. definitions, it is used in U. S. Government specifications to denote a definite specified stress which is to be applied to the specimen (or specimens) and then removed. If then the permanent set is less than a specified amount, the material is accepted; if not, it has failed. It is a "pass" or "fail" test.

In the second place, in the A.S.T.M. definitions there is a term already in use for a value obtained by the method Mr. Landon has used. That term is "yield strength."

He has, in fact, determined yield strength by the "total elongation" method as described in the A.S.T.M. standards.[2] He has reported values of modulus of elasticity which enable total elongation to be translated into terms of "offset" by the equation:

$$q = e - \frac{S_y}{E}$$

in which:

q = the "offset" from the straight (Hooke's law) line of the stress-strain diagram at stress S_y

e = the "total elongation" measured under stress S_y

E = the modulus of elasticity of the material; q and e are measured in inches per inch, S_y and E in pounds per square inch.

Mr. J. M. Lessells.[3]—I should like to associate myself with what Mr. Moore has said. The term "proof-stress" is adopted by the British Engineering Standards Association and is included in important British specifications, and, as Mr. Moore has said, it has not the meaning which the author gives it. Since it refers to the yielding of the material it is not related to the tensile strength but is more related to our term "yield strength."

Mr. Moore.—I think it is not always realized that the determination of the modulus of elasticity brings one over into almost one significant figure further of accuracy necessary than the determinations of yield strength. I am not proposing this for a standard but I would state that some years ago a very valuable *Bulletin* was published in the University of Illinois by Messrs. Seely and Putnam,[4] in which they reported the moduli of elasticity obtained with a 2-in. extensometer which had not been calibrated—and most extensometers do have somewhat serious errors in their reading unless they are calibrated. The Editorial Committee wrote out of that *Bulletin* all references to the determination of the modulus of elasticity, stating that with the sensitivity of 0.0001 in. they thought an 8-in. length necessary for an accurate determination of the modulus of elasticity.

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[1] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Standard Methods of Tension Testing of Metallic Materials (E 8 - 36), and Standard Definitions of Terms Relating to Methods of Testing (E 6 - 36), 1939 Book of A.S.T.M. Standards, Part I, pp. 761 and 776, respectively.

[3] Associate Professor of Mechanical Engineering, Engineering Dept., Massachusetts Institute of Technology, Cambridge, Mass.

[4] *Bulletin 115*, Engineering Experiment Station, University of Illinois, Urbana, Ill.

MR. L. B. TUCKERMAN.[5]—In looking over the paper I was interested in the modulus values reported. It would add to the value of the paper if the author would give more details about the strain gage and extensometer that he used in determining those modulus values. The only statement is that "an extensometer (one division corresponding to 0.0005-in. elongation)" was used and there is no statement about the type or method of using those gages. The values which are obtained for modulus of elasticity frequently depend very materially upon the type of strain gage, and sometimes on the way it is applied. Were there any specimens which had appreciable curvature? Extensometers of the Olsen type whose gage line extends diagonally across the specimen are likely to give erratic readings on specimens which are not straight. I am interested to know just how these values were obtained because the 33,000,000 value struck me as very high.

MR. R. D. LANDON[6] (*Author's closure*).—The author accepts Moore's and Lessells' criticism of the use of the term "proof stress." While the method of determining "yield strength" described in Section (*b*) paragraph 26, page 763 of Part I, 1939 Book of A.S.T.M. Standards is similar to that suggested in the original paper, the author does not believe that the use of a sensitive extensometer is desirable in routine commercial tests of malleable iron. He hesitated, therefore, to use the term "yield strength" unless the method described was followed explicitly. Nevertheless there does appear to be more justification for the term "yield strength," particularly if its use would forestall international complications.

Both Moore and Tuckerman have made pertinent observations on the determination of modulus of elasticity. The original report submitted by the author to Committee A-7 on Malleable Iron Castings contained complete descriptions of all equipment used and detailed outlines of all steps in the procedure. An Olsen strain gage (1 unit on dial equalling 0.000066 in.) was used on all specimens for which modulus of elasticity determinations were made. Each specimen was accurately punch marked in a jig before the strain gage was attached. The author does not attempt to explain the abnormal value of 33,000,000 psi. and would prefer to classify it as a mistake. Modulus of elasticity determinations on 8-in. specimens should be of interest and value.

As a matter of fact the modulus of elasticity determinations were incidental in this project. They were included as general information only. The object of the investigation was to determine stress-strain relations that might be of aid in commercial testing procedure.

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[5] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

[6] Professor of Civil Engineering, Southern Methodist University, Dallas, Tex.

PROPORTIONAL LIMIT TESTS ON COPPER ALLOYS

By Cyril Stanley Smith[1]

Synopsis

Since ordinary methods of obtaining axial loading in tension have been found unsuitable for precise tests, a new form of gripping device for 0.5-in. diameter threaded end specimens is described here. The specimen is locked into a head containing the ball through which the load is applied and which is optically adjusted to be exactly on the axis of the specimen. The specimen is maintained at 32 ±0.1 C. A high-grade commercial testing machine is used and calibration corrections applied by which the loads are proportional if not accurate to 0.1 per cent. Machine error exceeds extensometer error at about 35,000 psi. Two Tuckerman extensometers reading to 0.000002 in. on a 2-in. gage length or an averaging one of similar sensitivity give strain readings which permit deviation from proportionality to be plotted, from which the stresses corresponding to an offset of as little as 0.0002 per cent can be confidently read.

Annealed 70–30 brass rod of small grain size (0.008 mm.) had a "proportional limit" corresponding to 0.0002 per cent deviation from proportionality of 23,000 psi. Stressing this to produce a permanent set of only 0.006 per cent dropped the proportional limit to 14,500 psi. and further stressing up to 35 per cent permanent elongation left the proportional limit at 8000 to 10,000 psi. By annealing at 300 C. after stretching 10 per cent the proportional limit could be raised to 29,000 psi., but the gain was entirely lost on small overstrain. The engineering significance of these tests is not yet evident.

Permanent set after small straining of annealed materials is approximately equivalent to the offset from the initial modulus line. In cold-worked brass the offset is many times greater than the permanent set.

In order to promote the more intelligent engineering application of its materials, the American Brass Co. has undertaken to study the interrelation of stress and strain for a variety of copper alloys in various commercial treatments. Although high precision tests are unnecessary in control work and are inapplicable directly to the engineer's needs by virtue of the sensitivity of the results to prior treatment of the material, it was felt that the tests should be made with reasonably high precision in order to obtain a better understanding of the basic effects of composition, structure, and treatment upon the elastic and inelastic behavior of commercial copper-base alloys.

Numerous tests have been made using extensometers reading to 0.001 per cent on a 2-in. gage length and the technique is well established. On attempting to obtain a precision ten times this value, special technique had to be developed.

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[1] Research Metallurgist, American Brass Co., Waterbury, Conn.

Extensometers:

The Tuckerman extensometer is so outstanding in simplicity, reliability, and sensitivity that it seemed the logical choice for these tests. This instrument,[2] which is essentially a Marten's extensometer with improved optics rendering telescope position relatively unimportant, can be read by a vernier scale to 0.000002 in. or 0.0001 per cent on a 2-in. test specimen. It is necessary to use an instrument on each side of the test specimen to obtain the average extension under load. After long experience with the use of two instruments and only after loading had been developed to a point where reasonably accurate axial alignment was always obtained, an averaging instrument was made. This (Fig. 1) employed two horseshoes with knife edges 0.4 in. apart passing around the sample and each rocking between the surface of the test specimen on one side and the frame of the extensometer secured to the other side of the specimen by a knife edge at the other end of the gage length. To the side of one of these horseshoes was attached a Tuckerman roof prism with rotational adjustment and to the other a simple right angle prism with a tipping screw. A Tuckerman autocollimator measured the angle between the two prisms and hence the average of the extension on the two sides. Results obtained from this instrument have been indistinguishable from those using two Tuckerman extensometers. One-inch knife edges can be used to obtain a greater range without resetting. The frame of the exten-

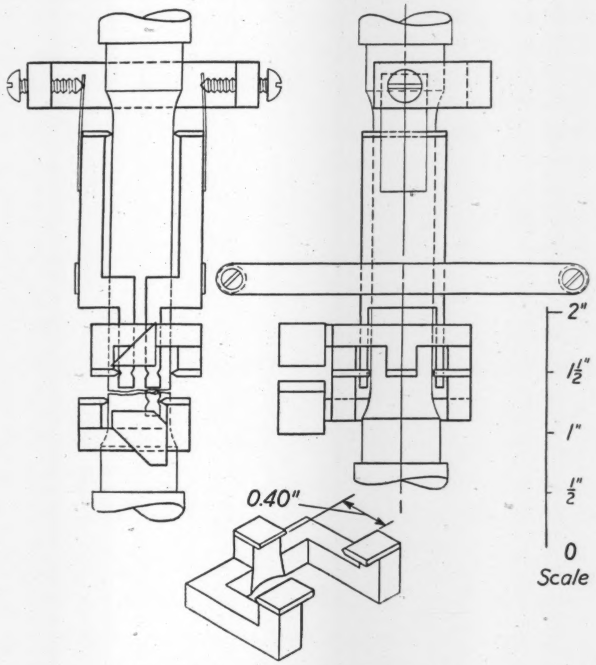

FIG. 1.—Averaging Extensometer.

The lower knife edge and prism are shown displaced to avoid confusion. The angle between the two prisms is measured with a Tuckerman autocollimator.

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[2] L. B. Tuckerman, "Optical Strain Gages and Extensometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 602 (1923).

someter is then reversed and the horseshoes rocked between the specimen surface and frame just as before. This device could, of course, be used with plane mirrors or right angle prisms but would then be affected by vibration and would require a fixed telescope position.

Testing Machine:

A Baldwin-Southwark testing machine made late in 1936 was used. It employs the Tate-Emery weighing system which involves the use of an Emery capsule and a Bourdon tube maintained at constant deflection by force applied through a pneumatically extended isoelastic spring. The machine has four ranges: 1000, 4000, 20,000, and 40,000 lb. It was purchased under a specification calling for an accuracy better than 0.5 per cent and with combined hysteresis and creep measured at mid range after 3 hr. at 95 per cent of capacity of less than 0.2 per cent. An error of 0.5 per cent in stress becomes greater than the extensometer error of 0.0001 per cent at a stress of 3400 psi. with a material having a modulus of elasticity of 17,000,000 psi. It was found, however, that correction factors from successive calibrations of the testing machine (made against proving rings and good to 0.1 per cent) remained fairly constant over a long period of time and such shifts as did occur were proportional to stress, that is, they were equivalent to a rotation of the correction curve around zero and hence affected the modulus but not the deviation from that modulus. All loads were therefore corrected on the basis of the calibration curve and under these conditions machine errors do not become important below stresses of about 35,000 psi.

Temperature Effect:

It is necessary to maintain the temperature of the specimen constant within 0.06 C. to avoid errors due to thermal expansion of the specimen greater than the extensometer sensitivity. A change of temperature affecting both steel gage and copper specimen equally can be as large as 0.25 C. without affecting the results.

The specimen and its holders were enclosed in an insulated box 14 by 19 by 32 in. fitting inside the uprights of the testing machine and containing a fan to circulate the air, which was maintained at 32 ± 0.1 C. by a mercury thermostat and two 50-watt heaters. The front of the box was removable to allow setting the specimen in place and contained a window for insertion of the autocollimator. The specimen holders required about 80 min. to reach temperature equilibrium, even though they had copper fins soldered to them. Temperature changes in the specimen resulting from elastic changes of stress were dis-

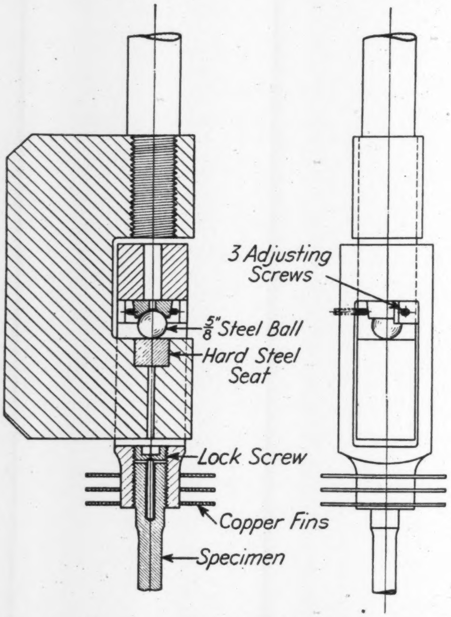

FIG. 2.—Specimen Holder Employed to Provide Axial Loading.

The threaded end of the specimen is seated by a force in the same direction as that subsequently to be applied in the test. The ball is adjustable to the axis of the specimen. Light springs (not shown) serve to center the holder on the seat at the commencement of the test.

sipated in 3 min. by conduction to the holders even under the most severe conditions.

Axial Alignment:

The specimen used for all tests was 0.505 in. in diameter with a $2\frac{1}{2}$ in. parallel section passing with 0.5 in. radius to 0.595 in. diameter, then to a $\frac{3}{4}$ in. threaded end to fit the holder.

Tests using the "self-aligning" holders with $1\frac{1}{2}$ in. radius spherical seats, as shown in Fig. 1 of A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8 – 36)[3] frequently showed differences in stress as high as 50 per cent between the two sides of the specimen at low stresses. Robertson ball-and-plane grips of the type shown in Fig. 3 of Method E 8 were found to be very little better because the fit between the threaded specimen and its holder was not perfect, and this fact entirely defeated the purpose of the device. A modification was accordingly constructed in which the threaded end of the specimen is first firmly seated in the threads of the holder by a force in the same direction as subsequently to be applied in the test, and the ball seat is then adjusted to be exactly on the axis of the specimen.[4] The device is shown in Fig. 2. The seating force from the locking screw is applied to the specimen by means of a stiff rod which passes down a hole drilled in the specimen head to a point just below the end of the threaded part. In mounting a specimen, the locking screws are tightened against the rods in the specimen while the heads are held in a jig to keep them roughly in alignment. Balls are held in place by temporary springs and the specimen is rested on two V supports 2 in. apart and rotated by hand. A telescope giving a magnification of about 4.5 is used to observe the image of a small source of light reflected in the back of the ball. Figure 3 shows the setup. As the assembly is rotated the point of light describes a circle the radius of which is equal to the eccentricity of the ball. Adjustment of the three screws on the seat permits the ball to be brought exactly to the axis of the specimen. Mounting and adjusting the specimen takes about 10 min. Except when using very small loads, new balls and newly polished hard steel seats are necessary for every test.

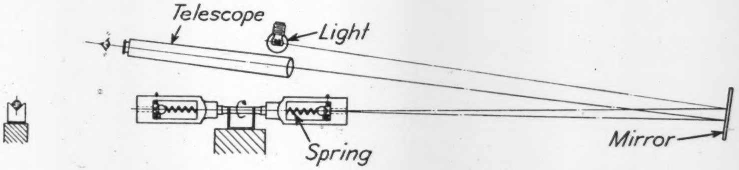

FIG. 3.—Optical Bench for Aligning Ball Seat on Specimen Axis.

With this device strain readings on two opposite sides of the specimen never differ by over 7 per cent and average about 3.5 per cent difference at an elongation of 0.1 per cent in 2 in. The differences are slightly greater at lower strains, averaging 6.5 per cent at 0.01 per cent extension.

Presentation of Results:

With the ordinary method of plotting stress-strain curves, deviations from proportionality less than 0.005 per cent are hard to observe regardless of the scale of plotting. It is, moreover, impossible to place the modulus line accurately through a series of such plotted points. By the use of the deviation method, apparently first proposed by McVetty and Mochel,[5] the full sensitivity of any extensometer can be utilized and the selection of the initial modulus usually becomes fairly definite. The points plotted are the difference between the observed values at a given stress and the values calculated from an assumed modulus. If the specimen is behaving elastically, the points fall on a straight line and this line is vertical if the actual and assumed modulus are identical. Sloping to left or to right indicates that the assumed modulus is lower or higher than the true one. It has been our custom to fit the first reasonable straight line to the points and to use as the proportional limit a deviation or offset from this corresponding to 0.0002 per cent. This will give lower figures than if a band of width ±0.0002 is applied to the experimental data.

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[3] 1939 Book of A.S.T.M. Standards, Part I, p. 750.

[4] M. Gensamer and R. F. Mehl, "Yield Point of Single Crystals of Iron," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 131, pp. 372–377 (1938).

Mr. Gensamer used an adjustable ball but located it by trial and error. The method of locking the specimen in the head and the method of optical alignment are thought to be new.

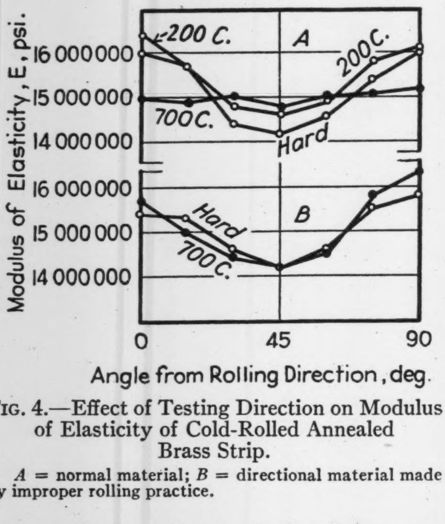

FIG. 4.—Effect of Testing Direction on Modulus of Elasticity of Cold-Rolled Annealed Brass Strip.

A = normal material; *B* = directional material made by improper rolling practice.

The strain readings not infrequently show a wave-like fluctuation around the elastic line. This can hardly be attributable to the behavior of the sample and may be due to variations of seating of the extensometer knife edges.

In spite of the high sensitivity in determining strain and deviation, the absolute values of stress are not so accurately known. Errors in the testing machine, in gaging the specimen, and in establishing the extensometer gage length combine to make possible errors in the absolute stress and modulus figures of about 0.5 per cent.[6]

Results:

The primary value of these high-precision tests is not at present so much to evaluate the materials in the engineer's sense as it is to throw light on the mechanism of the reaction of metals to stress. As an example of the type of result obtained some tests on brass rod are reported. The specimens were cut from a $\frac{3}{4}$-in. diameter cold-drawn rod containing 29.39 per cent zinc, 0.01 per cent lead, 0.01 per cent iron, balance copper. This rod was drawn to final size by a 65 per cent reduction of area following annealing to a grain size of

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[5] P. G. McVetty and N. L. Mochel, "The Tensile Properties of Stainless Iron and Other Alloys at Elevated Temperatures," *Transactions*, Am. Soc. Steel Treating, Vol. 11, pp. 73–92 (1927).

[6] A word of caution on the significance of an elastic modulus might be added. Any cast or wrought metal is somewhat directional and in copper alloys few properties are affected by crystal orientation as much as elastic modulus. By way of illustration Fig. 4 shows the variation of modulus with testing direction in cold-rolled and annealed 65-35 brass strip, 0.02 in. thick. These tests were made by E. W. Palmer. Specimens were held in a pin grip, with dead weight loading. Tuckerman extensometers were attached to each edge of a 2 by 0.5 in. gage length. Curve *A* shows normal commercial material cold rolled to 38 per cent reduction in thickness and also annealed 1 hour at 200 and 700 C. The modulus of the annealed material is identical in all directions. Curve *B* shows the modulus of some annealed material made following an undesirable rolling practice that left considerable directionality even after annealing. These data are introduced to show that in making a test in one direction only the modulus may vary over a considerable range depending entirely on the degree of directionality and that it may either increase or decrease on annealing depending entirely on orientation changes.

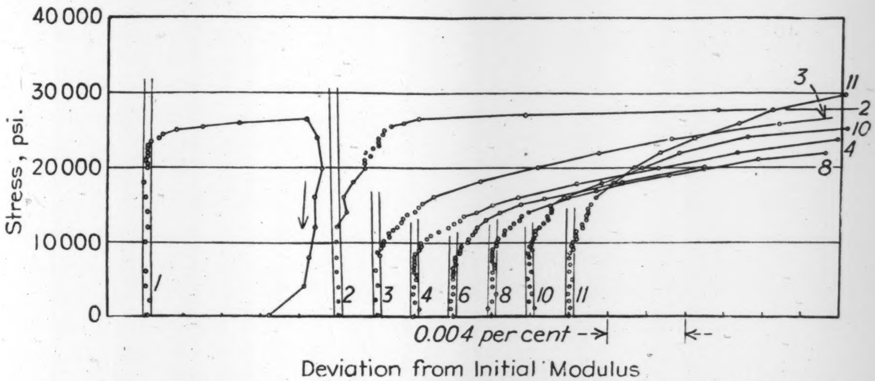

FIG. 5.—Stress-Strain Relations of 70-30 Brass Rod After Various Amounts of Prior Extension (Permanent Set).

Figures plotted represent deviation from trial modulus. All tests on single sample successively stretched to amounts shown. Each test based on new specimen diameter.

1 = 0.000 prior permanent set; E = 16 380 000 psi.
2 = 0.006 16 330 000
3 = 0.060 16 400 000
4 = 0.156 16 130 000
6 = 0.674 16 420 000
8 = 2.941 16 230 000
10 = 12.225 15 900 000
11 = 24.790 16 400 000

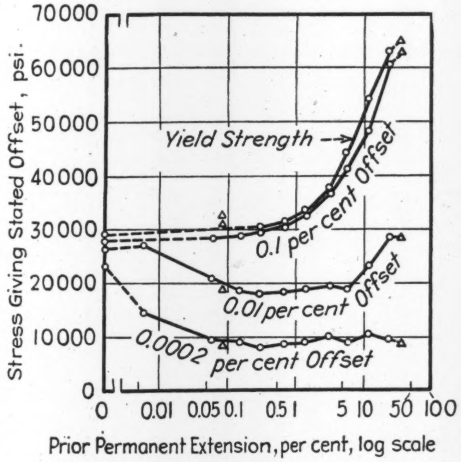

FIG. 6.—Effect of Prior Stretching on Stress Corresponding to Various Amounts of Offset from Proportionality.

Single specimen (cf. Fig. 5) progressively strained to give points plotted as circles. Triangles show results on different specimen stressed directly to 0.865 per cent and then to 35.0 per cent permanent set prior to testing with results shown. Yield strength corresponds to 0.5 per cent total extension under load.

0.060 mm. (This is not commercial treatment for brass rod but was given in order to permit the production of fine-grained material.) A sample was annealed for 2 hr. at 400 C. which produced a grain size of 0.008 mm. and was then strained in tension to various amounts of permanent set and retested to obtain the proportional limit after this treatment. The deviation curves are shown in Fig. 5. In Fig. 6 are plotted against the amounts of prior extension (permanent set) the stresses corresponding to various offsets during the next restressing. In the annealed material the stress corresponding to 0.0002 per cent offset was 23,000 psi. Stressing to only 0.006 per cent permanent set dropped this to 14,500 psi. and further stretching up to 35 per cent left it at 8000 to 10,000 psi. On the other hand, the stresses corresponding to deviations of a larger amount such as are ordinarily measured as yield strength or proof stress are at first unaffected and later raised by the prestraining treatment. Commercial cold-drawn material in which the direction of straining is not in the direction of subsequent tests has generally a higher proportional limit than shown in these tests and the reaction to tensile prestraining is different.

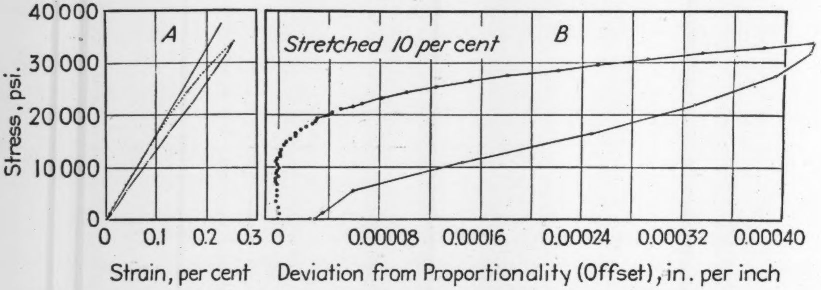

FIG. 7.—Stress-Strain Relations on 70–30 Brass Rod Previously Stretched to 10 per cent Permanent Set. Not annealed. Deviation from $E = 16,190,000$ psi.

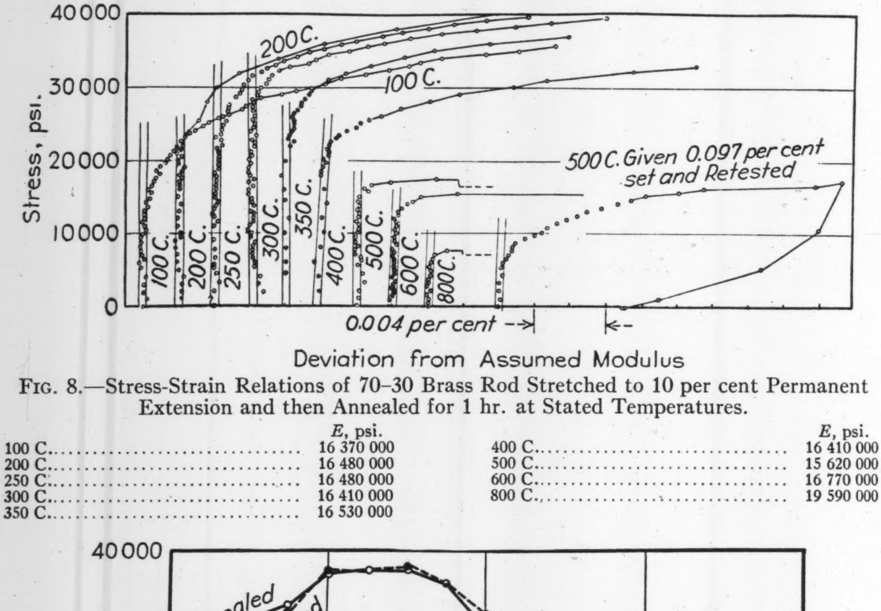

FIG. 8.—Stress-Strain Relations of 70–30 Brass Rod Stretched to 10 per cent Permanent Extension and then Annealed for 1 hr. at Stated Temperatures.

	E, psi.		*E*, psi.
100 C.	16 370 000	400 C.	16 410 000
200 C.	16 480 000	500 C.	15 620 000
250 C.	16 480 000	600 C.	16 770 000
300 C.	16 410 000	800 C.	19 590 000
350 C.	16 530 000		

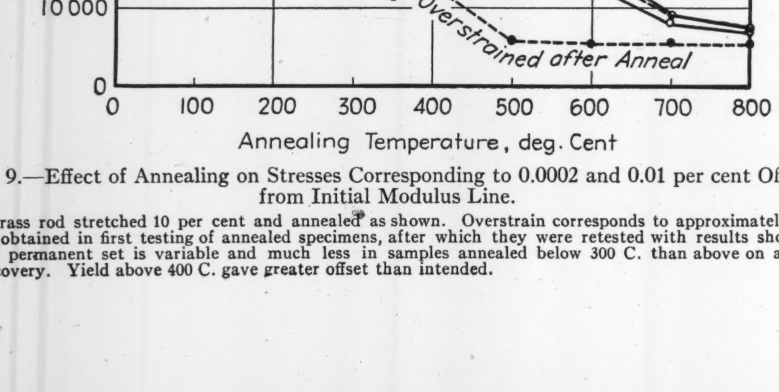

FIG. 9.—Effect of Annealing on Stresses Corresponding to 0.0002 and 0.01 per cent Offset from Initial Modulus Line.

70-30 brass rod stretched 10 per cent and annealed as shown. Overstrain corresponds to approximately 0.05 per cent offset obtained in first testing of annealed specimens, after which they were retested with results shown. The amount of permanent set is variable and much less in samples annealed below 300 C. than above on account of greater recovery. Yield above 400 C. gave greater offset than intended.

Figures 7 and 8 show deviation curves and the plotted results for a series of specimens of the same brass annealed at 400 C., stretched to 10 per cent permanent set, and then tested without annealing (Fig. 7) and after annealing 1 hr. at the temperatures stated (Fig. 8). Separate specimens were used for each curve. The stress-strain curve for each heat treatment was carried to an offset of about 0.05 per cent, the load released and a second curve obtained in this slightly over-strained condition.

Figure 9 shows the stress corresponding to offsets of 0.0002 per cent and 0.01 per cent plotted against annealing temperature. It also shows the same properties redetermined after the annealed specimens had been overstrained as described above.

Annealing at 300 C. causes complete recovery of the proportional limit and the brass is fully elastic to a stress of 29,000 psi. On the other hand, a small overstrain applied to this material lowers the 0.0002 per cent proportional limit almost to its initial value in the strained condition. The effect of small overstrains becomes less the higher the temperature of annealing above the recrystallization point, and suggests that the effect is largely associated with grain boundaries rather than the bodies of the grains. The stresses corresponding to 0.01 per cent deviation are also increased by annealing in the neighborhood of 300 C. and at any temperature are practically unaffected by slight overstrains.

Elastic Limit:

An "elastic solid" is defined in the Society's Tentative Definitions of Terms Relating to Rheological Properties of Matter (E 24 - 37 T)[7] as one in which strain is reversibly related to stress. This would seem to be a logical definition. On the other hand the property of "elasticity" requires only return to original size and shape after release of load, regardless of route traveled. The proportional limit seems to mark the limit of reversible elastic deformation, but beyond this there is still a range, at any rate in cold-worked materials, where irreversible or hysteretic elastic deformation is possible with complete recovery on release of load. This interpretation of proportional limit leaves out of consideration the fact that at very high stresses the line representing stress-strain behavior may perhaps be curved but reversible, just as compressibility varies at high pressures. Obviously this should be included in the elastic part of the curve but few copper alloys are elastic to sufficiently high stresses to show this behavior.

The curvature of the ordinary stress-strain curve that results from the fact that the dimensions of the specimen change under load is not negligible, as is commonly supposed, even in the elastic range. The difference between elastic behavior expressed in terms of true stress and true strain and the curve based on the original dimensions of the sample is, in terms of strain, approximately

$$\epsilon_0^2\left(\tfrac{1}{2} + 2\mu\right)$$

where μ is the true Poisson's ratio and ϵ_0 the observed elongation on the original length. The correction is unimportant below about 0.1 per cent elongation and has not been applied to the data in the present paper. With materials showing a greater elastic strain it is essential to make this correction if the departure from proportionality is to be evaluated. The stress-strain curves of tin bronzes and beryllium copper alloys show appreciable curvature attributable to this effect alone.

It is stated in Methods E 8 - 36[3] and in the Society's Standard Definitions of Terms Relating to Methods of Testing (E 6 – 36)[8] that the stress for a small permanent set is approximately the same as that for a deviation from proportionality of the same amount. This is far from true in the present tests. Figure 10 shows data to obtain the elastic limit of a sample of 70-30 brass annealed at 400 C. and similar data on the same sample after straining to a permanent set of 10 per cent. Deviation data are plotted for comparison. Each point of the set curve records the set after the whole series of stress cycles at progressively increasing stresses prior to this. Under these conditions it can be seen that agreement between set and offset is close in the annealed material, but in the strained specimen the stress producing a permanent set of any amount is much higher than the stress corresponding to the same degree of deviation from proportionality or offset.

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[7] 1939 Book of A.S.T.M. Standards, Part I, p. 1267; Part II, p. 1175; Part III, p. 1132.

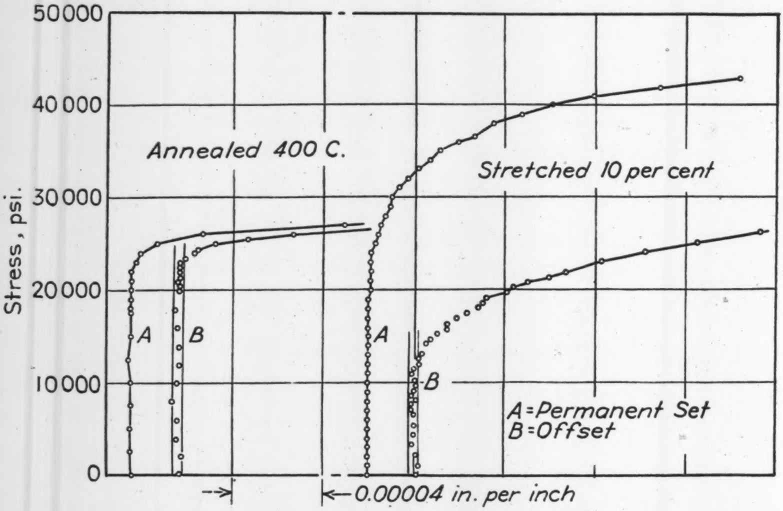

FIG. 10.—Comparison of Permanent Set and Offset from Modulus Line at Various Stresses.
70-30 brass cold drawn and annealed 400 C. (left) and the same material stretched to 10 per cent set (right).

The complete hysteresis loop, Fig. 7, shows the path of return from a high stress. This loop was the first restressing after the 10 per cent stretch. Note that the set is greater after this than after the same stress in the multiple cycle test. In another test when a specimen was taken at once to a load of 24,610 psi. and released a permanent set of 0.0010 per cent resulted, compared with only one vernier division (0.0001 per cent) from this stress when applied as a climax of a series of 24 cycles of gradually increasing stress. This indicates that the elastic limit as ordinarily determined is somewhat meaningless since the metal can be changed by the cycles at low stresses even though these produce no set.

Acknowledgments:

It is a pleasure to acknowledge the painstaking assistance of R. W. Van Wagner in developing the technique and making many of the measurements described in this paper. The author also wishes to record his indebtedness to the late H. C. Jennison who enthusiastically promoted the commencement of this work and to J. R. Freeman, Jr., Technical Manager of the American Brass Co., for his frequent advice and encouragement during its later stages.

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[8] 1939 Book of A.S.T.M. Standards, Part I, p. 776; Part II, p. 869; Part III, p. 551.

DISCUSSION

Mr. M. Gensamer[1] (*presented in written form*).—I am glad that Mr. Smith has pointed out how far from axial may be the loading obtained with the so-called self-aligning specimen holders in common use. We have had the same experience with threaded steel specimens and were forced to develop a modified Robertson shackle, as pointed out by Mr. Smith in the footnote in his paper. Mr. Smith's improvement eliminates the tedious trial and error adjustment and should be very much worth while, although we have found that with practice it becomes possible to secure alignment to within one part in thirty in about half an hour. To do this, we use four Tuckerman extensometers mounted at 90 deg. around the specimen and successively apply and release such a load as to get an extension of about 60 units on the autocollimator scale (total scale length, 2500 units). This method involves preloading to at least 1000 psi. (for brass). While this would ordinarily not affect the proportional limit, it certainly could do so with soft metals. Mr. Smith's method avoids this preloading and for soft specimens it or some equivalent would be absolutely necessary. I do not believe that it is practical to machine threads accurate enough, and specimens straight enough, to use threaded specimen holders without some such adjusting device when axial loading is necessary.

Mr. H. F. Moore.[2]—The standards of the Society covering elastic strength are written with the object of determining an index of resistance to structural damage of material by plastic action. Mr. Smith's paper evidently has other ends in view. This does not mean that it is of no significance; on the contrary consideration of delicate deviations from elastic action which are too small to cause direct structural damage may be of decided significance as an index of proper heat treatment or of other factors. As Mr. Smith states, at present the standards of the Society do not cover the determinations of such minute inelastic action at all adequately. This matter is already under consideration by the Section on Elastic Strength, of Committee E-1 on Methods of Testing, and Mr. Smith's paper will be of no small value to that section.

The present standards for yield strength speak of the practical equality of "offsets" (from the Hooke's law line) and "sets" for "small strains." The strains at yield strength are "small" when considered as structural damage, but are not small as compared with strains such as Mr. Smith is considering—say less than 0.1 per cent. The "offset" method cannot be regarded as useful for strains much, if any, less than this value.

I wish to thank Mr. Smith for helping us get the idea that below any known "true" elastic limit or proportional limit structural damage can be done to a metal by stress, strain, strain energy or shear-strain energy.

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[1] Metals Research Laboratory, Carnegie Institute of Technology, Schenley Park, Pittsburgh, Pa.

[2] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

Mr. E. E. Thum.[3]—Mr. Moore's remarks seem to imply that these results of Mr. Smith's could give some information about the early action of stressed copper around small discontinuities or other stress raisers. In other words, if you look upon ductility as a mechanism whereby the internal structure of a material adjusts itself, irons out and equalizes stresses, then these results by Mr. Smith may very well be capable of interpretation as to what happens specifically in the immediate vicinity of stress raisers in copper. If Mr. Smith *has* made any such interpretation, I hope he will give it to us.

I should like to ask whether any elastic action has been observed *after* the load has been released? In other words, is there any recovery with time? One of the diagrams shows a small permanent set existing after the so-called yield point had been passed by a very small load, and the entire load released. Does that set tend to recover with time, or do you know whether it does or not?

Mr. M. F. Sayre.[4]—Mr. Smith's paper deals with a subject that has been a hobby of mine for a number of years. About ten years ago I presented here a report on some measurements on brass and steel wires[5] in which we went to a sensitivity something like ten times greater than in this paper. When we reached that sensitivity we got into griefs, I think even beyond those which Mr. Smith has described here. He has avoided some of these difficulties by his lesser sensitivity and by delaying his measurement of elongation until after the initial creep and after the initial temperature equalization has occurred.

Even at figures well below the apparent proportional limit, when readings are taken immediately after application of load we find that the material shows a definite creep lasting for an appreciable period of time, how long I do not know, because the rate decreases on something like a logarithmic scale. Further, on removal of load, there is a plastic recovery of more or less the same amount. Then, too, the evidence to my mind is very strong that even in the first part of the elastic stress-strain curve, the line is theoretically not truly straight, that is, that inherently the behavior of the material is one of a changing modulus. The deviation is not large, but there are good physical reasons for believing that it is present.

I am very glad that Mr. Smith has called attention to the variations in the modulus of elasticity in different directions in the material which he has tested, and in particular that he has brought out the fact that to develop these variations, measurements must be taken in directions 45 deg. apart rather than 90 deg. Single crystal measurements, of which many have been made, particularly in Europe, show that for example in single crystals of brass, the tensional modulus of elasticity along the space diagonal between the crystal axes may be 3.5 times as great as when measured along any one of the three rectangular crystal axes. There is an inverse relation for the torsional modulus, the torsional modulus being high where the tensional modulus is low, and *vice versa.* With single crystals of iron similar relationships exist, the deviations here being of the order of two to one. Ordinary structural material is of course not in single crystal form, but any cold working is likely to develop a preferred orientation, with resulting directional properties. It seems to me that we must learn to recognize these large variations in modulus and along with this to avoid our very casual use of a standard figure, except where we know that we have an annealed material.

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[3] Editor, *Metal Progress,* American Society for Metals, Cleveland, Ohio.

[4] Professor of Applied Mechanics, Union College, Schenectady, N. Y.

[5] M. F. Sayre, "Elastic Behavior of Spring Materials," *Proceedings,* Am. Soc. Testing Mats., Vol. 30, Part II, p. 546 (1930).

MR. O. C. RUTLEDGE.[6]—I have had occasion to go over some of the data Mr. Smith has taken and compare it with similar data taken on duplicate specimens using a Sayre extensometer reading extensions to the nearest one thousandth of one per cent. This is only one-tenth of the sensitivity used by Mr. Smith and at this sensitivity the correlation is very good. The fact that Mr. Smith obtained so nearly a straight line in the lower section of his deviation curves is, I think, a very good indication of the accuracy of his work at that sensitivity. I should like to concur with the others who have discussed this paper; the study of this data is aiding us materially in understanding the elastic and plastic behavior of metals.

MR. L. B. TUCKERMAN.[7]—My name was taken in vain in this paper once or twice and I want to make a few comments on the optical lever system used in my strain gage. It is no more accurate or sensitive than the single and double-mirror systems such as used in the Martens and Sayre extensometers. It is merely more convenient because of the freedom of motion of the viewing instrument, the autocollimator. We have made the prism very small because for many uses it is desirable to have a small extensometer. I think it would be advisable in building averaging extensometers (extensometers which read directly the average of the extension on two or more gage lines) such as Mr. Smith has done to use a larger roof prism; it would give clearer images and easier reading. I am very much interested in this development. I have several times thought of making an averaging extensometer using my triple mirror autocollimated optical lever system but none of those I have thought of was anywhere near so simple in construction as Mr. Smith's. I wonder whether it could not be developed into a snap-on extensometer. If used exclusively in routine testing of one type of material, building the frame of the gage of material of the same temperature coefficient of expansion simplifies temperature control very much.

I was also interested to learn of the publication by McVetty and Mochel of the use of the deviation method. When I used the method some years ago in a discussion of a paper by Templin[8] I had not seen their paper and did not learn of it until Mr. Smith called it to my attention. It may be interesting to give a little of the history of the method. This is an adaptation of a method well known to physicists for many years. Where it is found that observational data follow approximately some definite mathematical law (not merely a straight line) and it is desired to study the deviations from that law, values accurately obeying that law are computed and differences between the observed and computed values are plotted. When small they can be plotted to an exaggerated scale so that they can be really studied instead of merely guessed at. As a good example of this more general use, which I happen to remember, I may cite the Reference Tables for Platinum to Platinum-Rhodium Thermocouples.[9,10] The National Bureau of Standards reference tables are regularly used by manufacturers in the calibration of direct reading thermoelectric thermometers. Many more citations could readily be found by a search of technical journals. It is my opinion that the method should be more widely used in studying stress-strain curves.

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[7] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

[8] Discussion by L. B. Tuckerman of paper by R. L. Templin, "The Determination and Significance of the Proportional Limit in the Testing of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 538 (1929).

[9] R. B. Sosman, *American Journal of Science*, Vol. 30, pp. 1-15 (1910).

[10] William F. Roeser and H. T. Wensel, "Reference Tables for Platinum to Platinum-Rhodium Thermocouples," *Journal of Research*, Nat. Bureau Standards, Vol. 10, February, 1933, pp. 275-287.

Incidentally, I wish to point out that the "wave-like fluctuation around the elastic line" which the author notes and is seen most clearly in Figs. 8 and 10 is not necessarily caused by any irregularity in the measurements. It is more probably what we call the "step effort"—a fairly common artificial effect occurring in accurate measurement where the constancy of functioning of the measuring instrument and closeness of control of the other experimental conditions is such that the least count error of the instrument is one of the major sources of error. If you are to use the deviation method to its best advantage, you must plot the deviations on a scale so large that the least count of the extensometer is readily seen. When that is done, the jump of the least count of the instrument is readily noticeable. If you take a smooth curve, a mathematically accurate curve, and compute the values close enough and then round them off to a certain number of significant figures, the difference curve will frequently drift over one unit in the last place only after two, three, four, five, or six readings; there will come the jump back, slide across and jump back again, giving the artificial appearance of a curve which has no relation to the behavior of the quantity measured. The limitation of the least count of the instrument causes the appearance. It has frequently been mistakenly interpreted as a real effect.

One other comment, and I am merely repeating what Moore has said; Mr. Smith has added much to our ammunition, when we try to persuade people that an offset is not a set. We know more about many materials now when we are measuring offsets of the order of one or two hundredths of one per cent. There is no necessary relation between these offsets and the permanent set of the material, after the release of the load. However, for the peace of mind of some of us more practical engineers who only occasionally can delve into these submicroscopic behaviors of materials, I wish Mr. Smith would reassure us that in these materials, if we are measuring offsets of the order of one tenth to two tenths of one per cent, we can be reasonably sure that the offset gives us a fair measure of the permanent set at offsets of that magnitude. I should hate to think that we shall be obliged in all of our specifications testing to release the load repeatedly and actually measure the permanent set in order to form a reasonable estimate of the load which would produce a permanent set of the order of one to two-tenths of one per cent. I wish that in preparing his reply, he would give us some data as to whether for these large offsets we can, as we have always hoped to do, reasonably use offsets instead of sets.

There is only one flaw in this otherwise extremely valuable paper that I have been able to find, and that is his use of the words "proportional limit." It seems to me that it is time that we should banish forever from the nomenclature of testing those two phrases, "proportional limit" and "elastic limit." We know there are no such limits in any of the ordinary polycrystalline aggregates which we use. If we need to specify the values these terms are used to represent, I would like to suggest that we speak of a "0.01 per cent offset stress" or a "0.01 per cent set stress." I doubt the practical value, except for very special uses, of stresses based upon such small deviations from Hooke's law, but I should like to see, instead of a "yield stress" which may not represent a yield at all, that we would speak, say, of a "0.2 per cent offset stress", or a "0.2 per cent set stress", specifically stating just what measurements we made and just what we found.

MR. A. R. ANDERSON[11] (*by letter*).—With increasingly precise measurements of tensile stress-strain relations, added care must be employed in interpreting the data. It has frequently been pointed out that in the range of plastic deformation the figures ordinarily used in plotting stress-strain curves, and which are referred to the initial cross-sectional area and initial gage length, do not represent true stresses and true strains. The errors from use of the nominal figures have been generally supposed to be negligible for the elastic part of the test. That this is not always the case is shown by the following considerations of the magnitude of the effect. This work was carried out at the suggestion of the author.

As a specimen elongates under load the gage length which the extensometer spans progressively increases; consequently, the actual unit strain ϵ is somewhat less than the figure ϵ_o obtained by dividing the total extension ΔL_o by the initial gage length, L_o.[12] An exact expression for strain is obtainable by considering an infinitesmally small increment of strain, $\frac{dL,}{L}$ integrated over the given range of extension. The true strain, ϵ, then becomes

$$\epsilon = \int_{L_o}^{L} \frac{dL}{L} = \log_e (1 + \epsilon_o)$$

$$= \epsilon_o - \frac{\epsilon_o^2}{2} + \frac{\epsilon_o^3}{3} - \ldots\ldots (1)$$

When a specimen is stressed in tension its cross-sectional area decreases. True unit stress, S, obtained by dividing the load by the actual area, A, is, consequently, somewhat greater than S_o, the value ordinarily calculated on the original area, A_o. It has usually been assumed that there was no volume change, whence

$$S = (1 + \epsilon_o)S_o \qquad (2)^*$$

Actually the volume change is appreciable and cannot be neglected. For a round cross-section

$$S = (1 + \epsilon_o)^{2\mu} S_o \cong (1 + 2\mu\epsilon_o) S_o \qquad (3)^*$$

Where μ is Poisson's ratio, here defined as the ratio of the true unit contraction to the true unit extension. This is positive in sign. In terms of t_o (the relative change in initial diameter, $\frac{\Delta D_o}{D_o}$, which is negative for a tension test, true stress is

$$S = \frac{S_0}{(1 + t_0)^2} \qquad (4)^*$$

From the preceding it is evident that at every point above the origin the usual stress-strain curve (S_o *versus* ϵ_o) lies somewhat to the right of and below the true stress-true strain curve (S *versus* ϵ) and that the higher the strain the greater becomes the separation between these curves. *If the true stress-true strain curve follows Hooke's law the ordinary stress-strain curve will be curved from zero stress* and a proportional limit determined from the latter will appear progressively lower as the precision of testing and scale of plotting the data are increased. Such a proportional limit obviously bears no relation to the point of actual departure from proportionality between true stress and true strain. The accompanying Fig. 1(*a*) shows this. The S_o *versus* ϵ_o curve has broken away from the straight true modulus line at extensions around 0.4 per cent. Figure 1(*b*) showing deviation from proportionality on a large scale clearly indicates the effect even below 0.1 per cent. It might be pointed out that if the

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[12] A complete list of symbols and meanings is appended, see p. 881. The accompanying Fig. 3 may assist in visualizing the various quantities.

* The derivations of the equations used in this discussion may be secured from Mr. Anderson upon request.

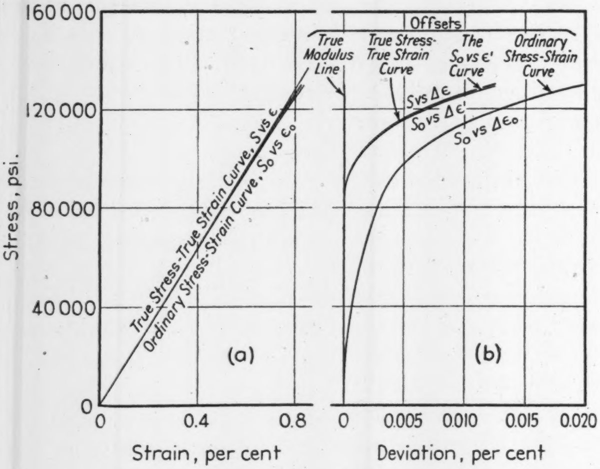

FIG. 1.—Relationship of Stress-Strain Curves.

(*a*) Comparison of a nominal stress-strain curve and the true stress - true strain curve. Hypothetical case for Poisson's ratio of 0.3, when the true curve is linear to 85,000 psi. stress and 0.5313 per cent strain and arbitrarily curved thereafter. Drawn to scale.

(*b*) Deviation from proportionality of curves of Fig. 1 (*a*) and of the S_o *versus* ϵ' curve which, in Fig. 1 (*a*) would be indistinguishable from the S *versus* ϵ curve. Note how well the true relationship is represented by the S_o *versus* ϵ' curve without corrections. Drawn to scale.

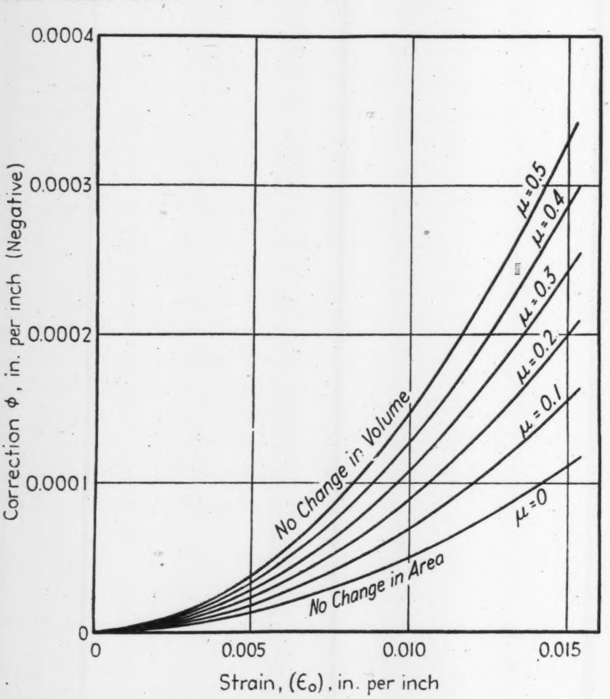

FIG. 2.—The Strain Correction, ϕ, Used To Obtain ϵ', Varies with Nominal Strain ϵ_o and Poisson's Ratio μ.

nominal data of Fig. 1 were plotted without a knowledge of the true modulus a straight modulus line of somewhat less slope might well be drawn making the deviation of the S_o *versus* ϵ_o curve in the elastic range considerably less apparent and indicating a false proportional limit.

The true stress - true strain curve can be constructed from ordinary S_o and ϵ_o data by correcting the stress figures to allow for the decrease in cross-section and correcting the strain figures for the progressive increase in gage length. The true value of stress for any nominal value S_o is obtained by adding $2\mu\epsilon_o S_o$ to S_o (see Eq. 3). This correction involves but an exceedingly small error from the neglect of higher order terms. The true strain for a nominal value ϵ_o is found by adding $-\dfrac{\epsilon_o^2}{2} + \dfrac{\epsilon_o^3}{3}$ to ϵ_o (from Eq. 1). Here, again, the correction involves a negligibly small error from neglected higher order terms.

The above straightforward procedure of correcting both stress and strain figures results in unequal increments of true stress and of true strain regardless of whether the nominal data are in equal increments of stress or strain. This complicates plotting of the corrected data and adds appreciably to the labor involved in obtaining curves showing deviation from proportionality (offset). When the experimental data are obtained in equal increments of nominal stress it is advantageous to add a single correction to each nominal strain figure which takes into account both the increase in gage length and the decrease in cross-sectional area.

A correction ϕ has been derived which when added to ϵ_o gives a strain figure ϵ' which is in the same proportion to S_o as the true strain ϵ is to the true stress S. If these values of ϵ' are plotted against the values of S_o for which they are obtained the resulting curve will represent the true stress - true strain behavior plotted to a distorted scale, the distortion being very small but increasing with strain. For most purposes the curve and its offset curve can be taken to represent the true stress-true strain behavior of the specimen.

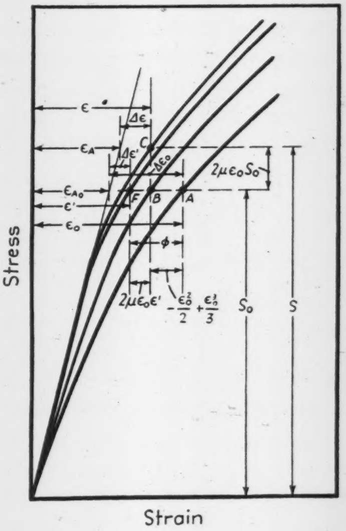

FIG. 3.—Illustration of Various Quantities Discussed.

Points *A*, *B*, *C*, and *F* are equivalent points on the several curves for the same load *P* on the specimen. *Not to scale.*

The strain ϵ' is given by the expression:

$$\epsilon' \cong \epsilon_o - (\tfrac{1}{2} + 2\mu)\epsilon_o^2 + [\tfrac{1}{3} + 2\mu(1 + \mu)]\epsilon_o^3 \qquad (5)^*$$

whence

$$\phi = \epsilon' - \epsilon_o \cong -(\tfrac{1}{2} + 2\mu)\epsilon_o^2 + [\tfrac{1}{3} + 2\mu(1 + \mu)]\epsilon_o^3 \qquad (6)$$

For small strains the ϵ_o^3 term can often be omitted and the correction, ϕ, to be added to the observed strain ϵ_o becomes

$$\phi \cong -(\tfrac{1}{2} + 2\mu)\epsilon_0^2 \quad \text{(Simplified expression)} \qquad (7)$$

A strain error, not exceeding 5×10^{-5} per cent results from using this simplified form for strains not greater than 0.75 per cent when μ is about 0.3. Tabulation or graphical representation of the correction for different values of ϵ_o and μ makes it possible to obtain ϵ' from ϵ_o by simple subtraction (ϕ being negative). The accompanying Fig. 2 shows, in a general way, how the correction varies. In practice this correction can be made at the time the offset is computed.

The accompanying Fig. 3 is included to interpret geometrically various symbols used in this discussion. It is obviously drawn all out of proportion for illustrative purposes and does not represent the various quantities in their relative magnitudes. All dimensions shown refer to a single condition (that is, applied load and resultant elongation) of the specimen, which would be plotted as point A from "nominal" data, as point B for true strain against nominal stress, as point C from true stress and true strain figures and as point F for the strain ϵ' against nominal stress.

As stated above the S_o *versus* ϵ' curve for most purposes can be considered identical with the S *versus* ϵ curve. However, if desired, a point on the S *versus* ϵ curve corresponding to any point on the S_o *versus* ϵ' curve can be found as follows: To the given value of S_o add $2\mu\epsilon_o S_o$ to obtain S (Eq. 3) and to the corresponding value of ϵ' add $2\mu\epsilon_o\epsilon'$ to obtain ϵ.* Deviation (offset) curves are handled similarly. The stress value is corrected, in the above manner, and to $\Delta\epsilon'$, deviation of the S_o *versus* ϵ' curve, for stress S_o, is added $2\mu\epsilon_o\cdot\Delta\epsilon'$, to give $\Delta\epsilon$, the deviation of the S *versus* ϵ curve for the corrected stress figure, S.* The factor $2\mu\epsilon_o$ enters into each of these corrections and is small. For a Poisson's ratio of 0.3 it does not exceed 0.006 for strains of 1 per cent or less.

As an example, if the S_o *versus* ϵ' curve showed a strain deviation of 50×10^{-4} per cent at a nominal stress of 90,000 psi. with a strain, ϵ', of 0.5467 per cent the S *versus* ϵ curve would show a deviation of 50.17×10^{-4} per cent at a true stress of 90,297 psi. and a true strain of 0.5485 per cent. If the stress, strain and/or deviation were less the corrections would be smaller. The factor $2\mu\epsilon_o$ in this case is 0.00330 indicating a correction of about one-third per cent in all three quantities. The value of true strain for a true stress of 90,000 psi. would be not far from the 0.5467 per cent figure obtained for ϵ' for a nominal stress of that value.

The curves of Fig. 1 are for a hypothetical case having a linear true stress - true strain relationship up to 85,000 psi. stress drawn to scale for a modulus of 16,000,000 psi. and a Poisson's ratio of 0.3. The deviation curves (Fig. 1(*b*)) clearly show how nearly identical the S_o *versus* ϵ' and S *versus* ϵ curves are; they cannot be separately identified when drawn to the scale of Fig. 1(*a*). It is evident from these curves that for small offsets if the stress values alone of a S_o *versus* $\Delta\epsilon'$ curve are corrected the resulting curve (S *versus* $\Delta\epsilon'$) is practically indistinguishable from the true (S *versus* $\Delta\epsilon$) deviation curve.

For ordinary purposes the difference between the nominal and true stress-strain curves is quite unimportant and indeed is less than the combined gaging, extensometer and machine errors. It is significant, however, when one is concerned with the validity or otherwise of

Hooke's law and with finding the point of departure from proportionality. Determination from an uncorrected curve of proportional limit corresponding to an offset of 0.0002 per cent may be subject to considerable error if the material is elastic much above 0.2 per cent strain. This can be seen by attempting to apply a straight line to a curve such as S_o *versus* $\Delta\epsilon_o$ of Fig. 1(*b*). It becomes impossible to find such a proportional limit if it occurs at an extension greater than about 0.3 per cent. Any possible curvature in the elastic portion of a stress-strain diagram should be considered only after making suitable corrections to the usual data. Omission of corrections is sufficient to account for much of the frequently observed bend in the elastic region.

Since none of the materials considered in the paper under discussion was elastic to strains exceeding 0.2 per cent, application of these corrections would not materially affect the observed values of proportional limits.

SYMBOLS USED

L_o = Initial gage length, no load.
L = Gage length under load P.
ΔL_o = Change in gage length from no load to load P. $\Delta L_o = L - L_o$
dL = Differential of L.
ϵ_o = Relative extension for load P, referred to initial gage length. $\epsilon_o = \dfrac{\Delta L_o}{L_o}$
ϵ = True longitudinal strain for load P, referred to continuously variable gage length. $\epsilon = \displaystyle\int_{L_o}^{L} \frac{dL}{L}$
ϵ' = A strain which is to nominal stress S_o as true strain, ϵ, is to true stress, S.
ϕ = A strain correction which, added to ϵ_o, gives ϵ'.
P = Axial load on specimen.
A_o = Initial cross-sectional area of specimen, no load.
A = Cross-sectional area under load P.
S_o = Nominal stress, computed on initial area. $S_o = \dfrac{P}{A_o}$
S = True stress, computed on actual area. $S = \dfrac{P}{A}$
D_o = Initial diameter of specimen cross-section, no load.
D = Diameter under load.
ΔD_o = Change in diameter from no load to load P. $\Delta D_o = D - D_o$
dD = Differential of D.
t_o = Relative change in diameter for load P referred to initial diameter. $t_o = \dfrac{\Delta D_o}{D_o}$
t = True lateral strain for load P, referred to continuously variable diameter. $t = \displaystyle\int_{D_o}^{D} \frac{dD}{D}$
μ = True Poisson's ratio. $\mu = -\dfrac{t}{\epsilon}$
E = Elastic modulus (assumed constant).
E_A = An assumed (constant) modulus for computing deviation data.
R = The ratio $\dfrac{\epsilon}{S}$ for all values (not a constant).
ϵ_A = Strain computed for a modulus E_A and stress S. $\epsilon_A = \dfrac{S}{E_A}$
ϵ_{A_o} = Strain computed for a modulus E_A and stress S_o. $\epsilon_{A_o} = \dfrac{S_o}{E_A}$
$\Delta\epsilon_o$ = Deviation of S_o *versus* ϵ_o curve from E_A modulus line for stress S_o. $\Delta\epsilon_o = \epsilon_o - \epsilon_{A_o}$
$\Delta\epsilon$ = Deviation of S *versus* ϵ curve from E_A modulus line for stress S. $\Delta\epsilon = \epsilon - \epsilon_A$
$\Delta\epsilon'$ = Deviation of S_o *versus* ϵ' curve from E_A modulus line for stress S_o. $\Delta\epsilon' = \epsilon' - \epsilon_{A_o}$
e = Base of natural logarithms.

MR. CYRIL STANLEY SMITH[13] (*Author's closure by letter*).—I should like to thank the various gentlemen who have discussed this paper for their valuable comments. Mr. Anderson's discussion will be useful to those who are concerned with the fine points of elastic and inelastic behavior. It remains to be seen how much curvature of the true elastic line exists when corrections such as these are applied.

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It is true, as Tuckerman says, that the optical lever system of his extensometer is basically no more sensitive than other types. Nevertheless, the instrument is considerably more accurate under ordinary conditions of use because it is unnecessary to maintain exact adjustment of the relative positions of the autocollimator and the prism arrangement. I do not believe the use of a larger roof prism is necessary. The restriction of aperture caused by the small lozenge in the standard extensometer is apparently responsible for the slight fuzziness, and the image is as clear and sharp as could be desired when the lozenge is replaced by the $\frac{3}{8}$ in. right-angle prism, as shown in Fig. 1.

The use of the deviation method of plotting stress-strain data is even older than the 1927 reference quoted in the paper. Batson and Hyde in Vol. I of their *Mechanical Testing*,[14] published in 1922, describe and illustrate the method but give no reference to its origin. The wave-like fluctuation of the data commented upon by Mr. Tuckerman seems to be too great to be accounted for by the least unit step of the instrument.

Mr. Tuckerman criticizes the use of the words "proportional limit" and "elastic limit." The author readily admits that these terms by themselves are meaningless but contends that when these are followed by a statement of the permitted offset or permanent set they represent a measurable property of matter and provide a figure of some use. Proportional limit defined in this way is of the same nature as the yield strength at the usual 0.2 per cent offset, but while the "proportional limit" indicates the first practically detectable plastic deformation the yield strength is meant to represent deformations that sometimes at least could be permitted in practice. There is danger, of course, that the term "proportional limit" will be used without a corresponding strain offset, in which case it is practically meaningless. Proportional limits determined from stress-strain curves plotted in the customary fashion are also of little value not only because of the difficulty of deciding where deviation first commences but more because of the difficulty of unequivocally establishing the initial modulus line.

Both Sayre and Thum mention creep and recovery effects. When using a specimen of the size described, the thermal effect resulting from change of temperature by adiabatic elastic expansion is dissipated by conduction to the massive specimen holders and does not cause any effect on slow application and release of load. The effect becomes visible in determinations of elastic limit but can be minimized by applying and removing the load as rapidly as is consistent with accuracy, for heat transfer between specimen and its surroundings is then small in either direction. If the load were maintained for several minutes to allow the specimen to reach thermal equilibrium under stress, then on releasing the load quickly the resultant heating of the specimen caused an expansion. For example, with heat-treated beryllium copper at a maximum stress of 90,000 psi. this amounted to 11×10^{-6} in. per inch but it was dissipated in 3 min. and there was thereafter no detectable change. When the same load was applied in 38 sec., held just 5 sec. and released, the thermal effect amounted only to 4×10^{-6} in. per inch and was completely dissipated in 2 min. These effects would be different with materials of different conductivity, but in every case the desirability of not holding the load for prolonged periods is evident. These comments relate only to small length changes resulting from the temperature change accompanying elastic deformation. With large amounts of plastic deformation there is a superimposed effect of more or less straight creep, that is, plastic elongation with time under constant load and temperature, and a partial reverse recovery at zero stress. This effect has not been studied in detail as it is not significant in the tests involving only minute plastic deformations.

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[14] R. G. Batson and J. H. Hyde, "Mechanical Testing," Vol. I, "Testing of Materials of Construction," Chapman and Hall, Ltd., London (1922).

Sayre suggests that the stress-strain curve is not necessarily straight even in the elastic region. I share his belief but feel that such curvature becomes detectable only in those few materials wherein very large elastic deformations are possible. It is mentioned in the paper that appreciable curvature results from the changes of dimensions of the specimen under load, and it is only after the corrections proposed by Mr. Anderson are made that the change of true modulus can be seen.

One specimen that had been strained 10 per cent, from the series that was annealed to provide the results shown in Fig. 8, was preserved at room temperature for 209 days (about 7 months) and has now been tested. The proportional limit (0.0002 per cent offset) was found to be 19,500 psi. indicating that the recovery effect is appreciable at room temperatures.

In the paper the comparison is made between offset and permanent set for 70-30 brass and it was shown that these two indices of plastic deformation are by no means equivalent. To answer

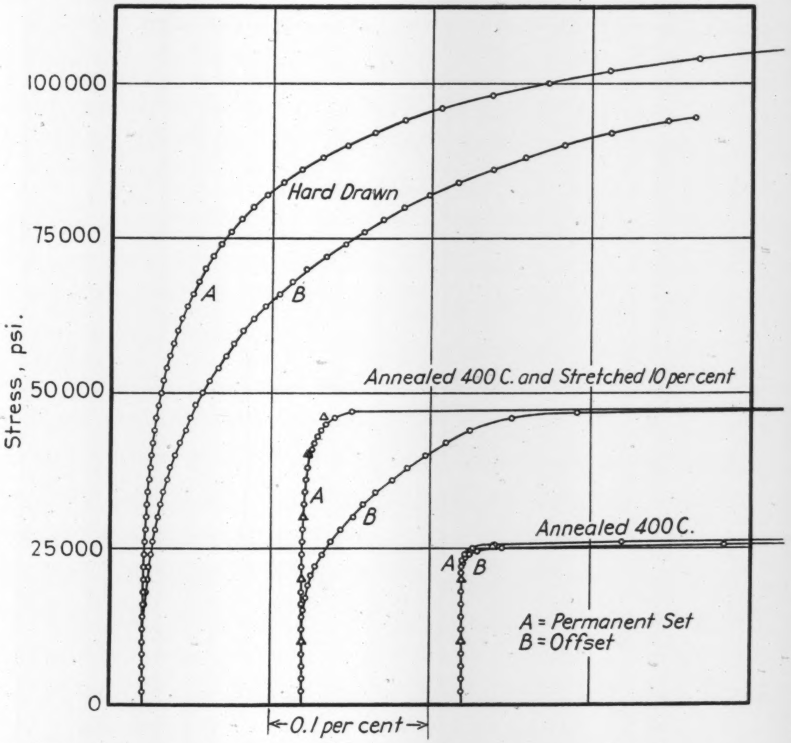

FIG. 4.—Comparison of Permanent Set and Offset at Various Stresses.

the comments of Moore regarding the present standards for yield strength, some additional information has been collected. The accompanying Fig. 4 shows data similar to that in Fig. 10 of the paper but continued to much larger amounts of strain. In the case of the material annealed at 400 C. and also when stretched in tension 10 per cent the stress corresponding to 0.2 per cent offset is about the same as that corresponding to 0.2 per cent permanent set. However, in the hard-drawn rod the stress corresponding to a 0.2 per cent offset is 84,500 psi. which produces a permanent set of only 0.089 per cent, and a stress of 97,000 psi. is needed to produce a permanent set of 0.2 per cent. This difference is not negligible even in commercial testing. Incidentally, the points plotted as triangles in the accompanying Fig. 1 represent the permanent set obtained by applying stress in large increments while the circles represent small increments. It is apparent from these that the difference between the two cases is not as great as implied in the paper.

INTERNAL HYDRAULIC BURSTING TESTS OF LEAD CABLE SHEATHING

By Howard S. Phelps,[1] Albert M. Gates,[1] and Frank Kahn[1]

Synopsis

One of the most vulnerable components of lead-sheathed power cable appears to be the lead sheath itself. This paper describes a method for making reproducible hydrostatic bursting tests of cylindrical lead specimens, which has been developed to simulate service conditions. Reliable test data are now being obtained from which it is hoped to determine the kind of lead or lead alloy which will best withstand internal pressures developed in service. Results to date have revealed information about lead sheathing that gives a new conception of the problem. Suggestions are made for further study.

Background

A method of testing lead cable sheathing that would give consistently reproducible results has been a need of long standing of both cable manufacturers and users. It is felt that developments in quality of lead sheathing and the use of advantageous lead alloys has not kept pace with improvements in design of the rest of the cable. This lag has been due in part to a lack of sufficient reliable data on which to base improvements. After experimenting with a number of the more commonly used methods of lead testing, the authors have developed a procedure which is simple in operation, gives reproducible results, and has promise of indicating relative quality of sheathing.

The importance of the sheathing problem became apparent about a decade ago when it was observed in Chicago, Philadelphia, and other places, that a large number of 69-kv. cable failures were caused by openings or splits in the sheathing of solid-type cables. These split sheathings were apparently caused by internal pressure resulting from loading cycles and the relatively new practice of maintaining positive oil pressure in the cable. No trouble of this kind was experienced with three-conductor 13-kv. cable at that time, or subsequently. Apparently this cable has ample fillers to compensate for expansion.

As a result of these experiences, general interest was aroused in methods of testing lead cable sheathing. As early as 1928 the Utilities Research Commission in cooperation with the Engineering Experiment Station of the University of Illinois started an extensive investigation of the creep of lead and lead alloys. The Insulated Power Cable Engineers Association formed a special committee in 1931 to analyze the situation, and was later joined by the committee on High-Tension Cables of the Association of Edison Illuminating

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[1] Engineer, Special Tester, and Senior Technical Assistant, respectively, Special Investigation and Testing Division, Philadelphia Electric Co., Philadelphia, Pa.

Companies. These committees inaugurated series of tests among member companies designed to show relative results of several of the more commonly used tests as conducted in different laboratories. Other investigations were made independently by both manufacturers and utilities.

In order to understand properly developments, it is necessary to be familiar with the early theories and practices in testing lead, in the form of fabricated cable sheathing as well as for the physical characteristics of the metal. It is especially important to be familiar with the effect of the welds on the sheathing.

Description of Welds:

With the lead presses in common use in America, welds occurring in the manufacture of lead cable sheathing are of two kinds, most conveniently classified according to their origin. The first of these classifications is the die weld. As the slug of lead is forced through the lead press most commonly used, it is divided into two streams by the bridge of the die block. The two streams then fold around the cable core and reunite at the top and bottom of the cable, forming two die welds. In a cross-section of cable sheathing, die welds take the form of practically radial lines extending from the inner to the outer surfaces of the lead.

The other classification is the charge weld. These are formed in the charging cylinder of the press between successive charges of molten lead. The charge welds are variously referred to by cable manufacturers as flow lines, charge loops, or tongues, when their effect is made evident in the sheathing. They appear in sheathing when that part of the lead slug including the charge weld goes through the die block and folds around the cable core. Since the slug cools from the outside, lead in the middle of the slug remains more plastic than the outside and hence is moved more rapidly by pressure from the press. The effect is that of a tongue of lead from the new charge penetrating into the remainder of the old charge. This tongue is divided by the bridge of the die block and appears as a tongue on each side of the cable. Successive cross-sections of the sheathing, then, contain series of loops increasing in size from points where the tips of the tongues first appear, to loops whose edges coincide with the die welds and the surfaces of the sheathing.

The section of sheathing between the first and last appearance of the charge loop is known as the critical region of the extrusion cycle. Under some operating conditions it is possible for two or more charge loops to occur in one cross-section. Usually there is a relatively long section of sheath known as the noncritical region containing, substantially, lead from only one charge.

Although these weld areas are theoretically present in all cable sheathing extruded with the type of press under consideration, it is sometimes practically impossible to detect them with the etching test when the more modern manufacturing techniques are used. A description of the more modern lead presses is beyond the scope of this paper, but a brief summary of them is given in an article by W. H. Bassett, Jr. (**1**),[2] and greater details in articles by Atkinson (**2**), Faucett (**3**), Peirce (**4**), Sherman (**5**), Reinitz (**6**), and Zickrick (**7**). These articles also include micrographs showing the weld areas mentioned above.

Little was known of the stress and strain characteristics of lead cable sheathing when the advent of high-voltage cable to transmit large blocks of power emphasized the seriousness of the problems associated with it. However, each of the cable manufacturers had his own system of testing to determine whether his product was of acceptable quality. The principal causes for rejection, other than eccentricity, were defects in either die or charge welds.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 903.

Weak die welds may result if proper conditions of temperature and pressure are not maintained at the die block. When the two streams of lead meet after being divided by the bridge of the die block, the temperature must be such that the pressure exerted will cause the lead to weld firmly together. If the lead is too cold, the two streams will simply be pushed together without welding, or with only partial welds. The result is known as a "cold weld".

The same problem obtains with respect to uniting two successive charges in the charging cylinder. In this case, the lead of the new charge is in a molten state and tends to heat the remainder of the old charge enough by contact to form a weld. However, there is the possibility that the weld will not be complete, in which case there will be discontinuous sections of lead in the press. There is also the possibility that oxides or other contaminating material will collect on the surface of a charge, and unless precautions are taken to eliminate or remove them, successive charges will be separated by a layer of inclusions. When this part of the slug is extruded on the cable core, the contaminating materials are included along the flow lines. Considering a longitudinal section of the sheath through the critical region, a condition of layers of separated lead known as "laminations" is created. The term is also applied to separated layers of lead without inclusions.

Tests for Integrity of Sheathing:

The early tests of the manufacturers were largely confined to detecting "cold welds" and "laminated lead." Of necessity, they were rapid tests that could be performed in the factory without unduly delaying production. Among those used were the bending test, drawbench test, and several variations of a test that expanded ring specimens. These tests are described in I.P.C.E.A. publications (8, 9).

In the bending test, "cold welds" were detected by the radial nature of the break, following the die weld from the inside to the outside of the sheath. Lead with excessive inclusions was disclosed by rounded breaks which followed the curve of the charge loop from the inside to the outside of the sheath in much the same manner as the radial break; or by the nature of the laminated breaks where the fracture traversed the several layers of lead formed by the flow lines. While radial and rounded breaks were generally accepted as representing defective sheathing, the seriousness of defects causing laminated breaks was largely a matter of the opinion of the person making the test. If a large amount of inclusions was present—detected by granular appearance of the lead—the sheath was apt to be rejected. If, however, only a moderate amount of inclusions was found, or simple separations of layers of lead, the tendency was to accept the sheath as sound.

This tolerance was influenced by results of stretch tests on ring specimens. These tests were favored when time permitted because they more closely resembled conditions obtaining in the field. In these tests, defective welds were revealed by radial or rounded breaks similar to those obtained with the bending test. However, if the specimens contained only clean laminations or a moderate amount of inclusions, the lead would neck down to the same kind of a wedge-shape break that was obtained for homogeneous lead. The assumption was that for operating purposes the strength of sheathing was not impaired by laminations with moderate amounts of inclusions.

When the integrity of lead sheathing was questioned and cable users as well as manufacturers became interested in testing methods, an attempt was made to convert these tests for acceptability into tests for relative quality. This made it necessary to standardize test procedures and develop some system of index numbers for purposes of comparison. For example, the bending test procedure was standardized, and an attempt was made to use the number of bends to fracture as a criterion, as well as the type of break.

In conjunction with deformation tests, it became common practice to prepare an etched specimen of an adjacent section of sheath to show the relation of fracture to grain structure. Comparisons of these data supported the conclusion that both the bending test and various forms of the stretching test would detect "cold welds" and exceptionally bad "laminations." They also appeared to support the theory that in cases where a wedge-shape rupture was caused by any of the various forms of stretching tests, the strength of the lead was not seriously affected by the presence of laminations or small amounts of inclusions. However, there remained a wide band between sheathing generally accepted as bad and that which was incontestably good. Interest was intensified in classifying sheathing falling in this band.

TESTS BY THE PHILADELPHIA ELECTRIC CO.

Although the seriousness of the sheathing problem was recognized by the Philadelphia Electric Co. as early as 1931, it was not until the latter part of 1932 that delay in general progress prompted active participation in studies to solve it. It was realized that the study of creep was being ably covered by Moore at the University of Illinois, but it was not definitely established whether lead of low or high creep characteristics was preferable for sheathing. However, it was obvious that sheathing should not fail under operating pressures during the life expectancy of the cables. Accordingly, the most direct approach appeared to be a study of the strength of cable sheathing as opposed to creep.

After experimenting with some of the more commonly used tests, the authors concluded that the one having greatest promise consisted of applying internal pressure to a section of sheathing until failure occurred. This opinion was strengthened by the early reports of investigations by the I.P.C.E.A. committee (8) and the University of Illinois (10). Both of these investigations made approaches to the problem of applying stresses as they actually occurred in the field, but their main efforts were in other directions.

The principal objection to internal pressure tests was the lack of simplicity in conducting them. In spite of this the authors felt that the superior results obtained warranted more careful studies as to simplification of method and interpretation of data. As progress was made it was visioned that a correlation between long-time and short-time tests might be obtained, so that a normal life-expectancy of cable sheath could be forecast.

The present method of conducting constant pressure hydraulic bursting tests is the result of several converging branches of investigation. Although some of the results reported in this paper were obtained by earlier methods only the current one will be described.

Calculation of Stress:

In the early stages of the investigation it was realized that calculation of stresses in a sealed-end specimen was far removed from the case of a strip specimen in simple tension. A sealed-end specimen is subject to forces in tension in the circumferential and longitudinal directions and a force in compression in the radial direction. It was felt that the result of these forces should be known before conclusions could be drawn from test results.

Although other investigators have used the conventional hoop stress formula, $S = PD/2t$, it was felt that this approximation might not be adequate for sealed end specimens. Accordingly a formula was derived, using as a basis the theory set forth in Chapter 15 of "Mechanics of Materials," eleventh edition, by Mansfield Merriman. A value of 0.45 was used for the factor of lateral contraction (Poisson's ratio). This formula involves several assumptions and approximations, but it is felt to be as accurate as is warranted by the accuracy of the data on which it is based.

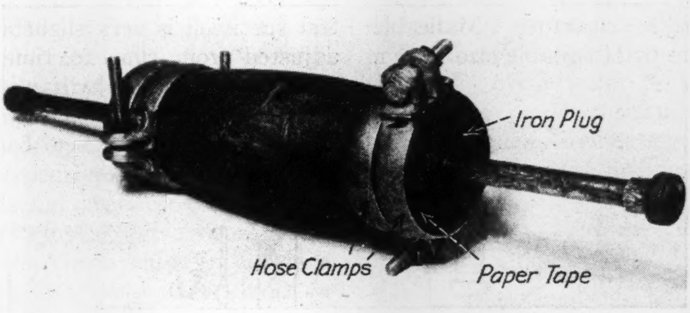


FIG. 1.—Method of Sealing Ends of Specimen with Hose Clamps.

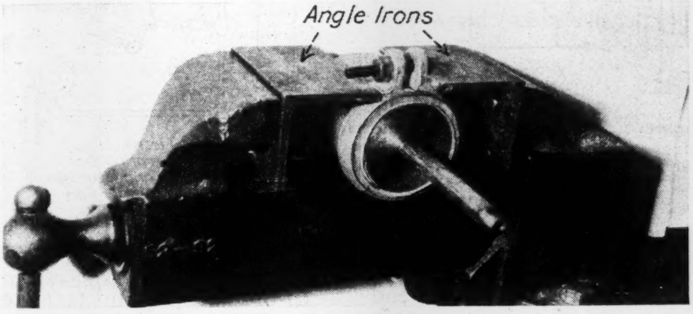


FIG. 2.—Use of Vise to Tighten Hose Clamps to Seal Ends of Specimen.

The formula for maximum stress is stated as follows:

$$S = PD/2t_{\text{min.}} + P\,(1.065 - 0.1125\,D/t_{\text{avg.}})$$

Method of Preparing Specimens:

A typical specimen prepared for test is shown in Fig. 1. Specimens approximately 12 in. long are used. The end plugs, shown in position, are solid iron cylinders 2 in. long. One of them is drilled through and tapped for a

$\frac{1}{4}$-in. pipe nipple for connection to the pressure supply. The other is drilled part way through and tapped for a piece of pipe to be used as a handle.

End plugs are selected slightly smaller in diameter than the inside diameter of the specimen. The diameters of the plugs are built up with ordinary gummed wrapping paper until they slide into the ends of the specimen without appreciable clearance. Malleable iron hose clamps of suitable size known to the rubber trade as "King" clamps are then slipped over the ends of the specimen and the clamps tightened, compressing the lead around the end plugs. A simple method of tightening the clamps in an ordinary vise is shown in Fig. 2.

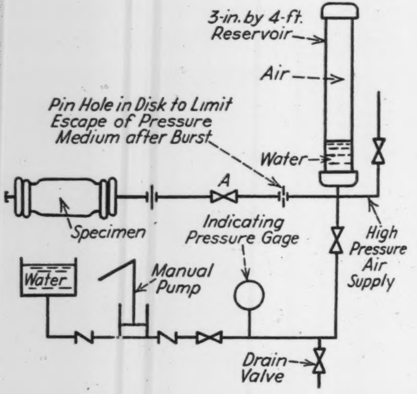

3-in. by 4-ft. Reservoir
Air
Pin Hole in Disk to Limit Escape of Pressure Medium after Burst
Water
A
Specimen
Indicating Pressure Gage
High Pressure Air Supply
Water
Manual Pump
Drain Valve

FIG. 3.—Air Reservoir Method for Maintaining Pressure on Specimen.

Method of Applying Pressure:

The method for applying pressure to the specimen consists of using a vertical pipe accumulator or reservoir, with an air cushion over a column of water. Suitable apparatus is shown diagrammatically in Fig. 3. With valve *A* closed, the required pressure is built up in the reservoir, using only as much water as is necessary to supply the specimen during expansion. This is advisable to prevent an explosive rupture of the specimen when it fails. The specimen is filled with water and connected to the pressure system by means of the union shown. Valve *A* is then opened and full pressure quickly applied to the specimen. With this arrangement, the change in pressure in the air reservoir due to the expansion of the test specimen is very slight and can be adjusted from time to time with the manually operated hydraulic pump.

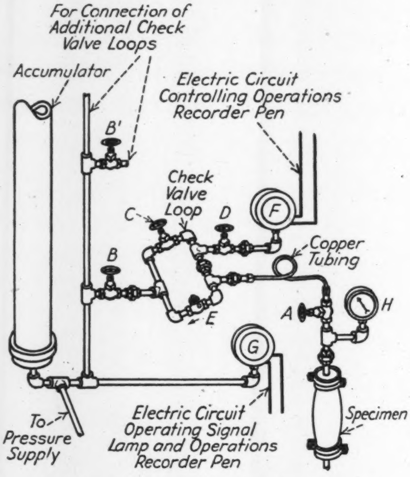

For Connection of Additional Check Valve Loops
Accumulator
Electric Circuit Controlling Operations Recorder Pen
B'
Check Valve Loop
C
D
F
Copper Tubing
B
E
A
H
G
Specimen
To Pressure Supply
Electric Circuit Operating Signal Lamp and Operations Recorder Pen

FIG. 4.—Diagram of Method for Making Multiple Tests with Single Accumulator.

In order to expand test facilities with the minimum equipment, means were devised for using a single accumulator to make simultaneous tests on several specimens at the same pressure. This is accomplished by providing each specimen with a check valve system to preserve pressure on the other specimens after any one bursts.

The apparatus is shown schematically in Fig. 4. Any number of specimens can be tested from one reservoir as indicated by valves *B* and *B′*. The essential feature for multiple operation is the swing check valve *E*, connected in a direction to oppose flow toward the specimen. The check valve is inclined from the horizontal so that gravity holds the valve disk free of the seat when the pressure is equalized. With the specimen connected as indicated and with starting valve *C* closed, the slight flow of water for normal expansion of the specimen does not close the check valve. However, when the specimen bursts, the check valve is closed by the pressure differential.

With this apparatus, some precautions are necessary to assure that full pressure is maintained on the specimens. Gage *H* is an inexpensive indicating pressure gage connected between the check valve and specimen to give visual assurance that the check valve is not in a closed position. Gage *G* includes a mercury switch which operates a low-pressure signal in the event that the check valve leaks after a specimen bursts, thus releasing the pressure on other specimens on the same reservoir. Gage *F* operates another signalling device that records the time of failure of the specimen to which it is connected.

Temperature Control:

The first tests were conducted at ambient temperatures. In order to reduce divergence of results, a thermally insulated box was constructed and equipped with thermostatic control for 110 F. The specimens are now suspended in this box and connected to the pressure supply with copper tubing.

A few tests have also been conducted in other boxes held at 150 F. and 50 F. With the present capacity, simultaneous tests can be made on 16 specimens at 150 F., 24 at 110 F. and 6 at 50 F.

In order to eliminate slight variations in pressure in the reservoirs due to ambient temperature changes, present plans call for enclosing the 8 reservoirs in use in a constant temperature compartment.

Cyclic Pressure Tests:

A point of general interest has been whether constant-pressure tests were comparable to field conditions, because of the known variations in internal pressure in cables in service. The apparatus shown diagrammatically in Fig. 5 was designed to investigate this point.

The theory of operation is that during the "off" cycle water is kept in the accumulator under a vacuum. During the "on" cycle, the heating coil creates steam and raises the pressure to the desired value. The pressure control gage and the auxiliary relay maintain this pressure within 1 lb.

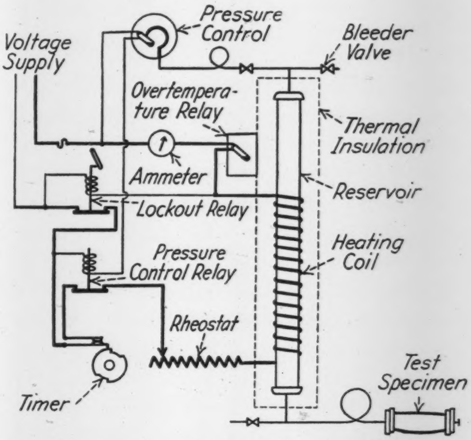


FIG. 5.—Diagram of Cyclic Pressure Apparatus.

The frequency and duration of "on" cycles are controlled by an automatic timer. The vacuum is originally obtained by bleeding steam during an "on" cycle before the test specimen is connected. The rate of increase of pressure is a function of the heating current. Since the heat generated may be a source of danger, especially after the specimen bursts, a safety device is installed, consisting of a thermostat clamped on the outside of the asbestos pipe covering. This is set to actuate a lockout relay which opens the heating circuit if the temperature rises to a predetermined value.

The present installation is capable of maintaining pressure up to 65 lb. safely. A vacuum of approximately 28 in. is possible.

RESULTS OF TESTS

The relative merits of step-pressure and constant-pressure tests were given a great deal of attention. A number of tests were made on specimens of sheathing as nearly identical as it was possible to obtain, using both methods, with results all in favor of constant pressure. Not only were the bursting times for the same stresses in reasonably close agreement, but the relation of time-to-burst and applied stress appeared to be consistent. On the other hand, reproducibility with the step-pressure method was very poor.

Although the method of constant-pressure tests was adopted, due consideration was given to the advantages of making tests on the basis of constant stress. There is an appreciable difference in the approach for cylindrical specimens under constant pressure, and tension test specimens under constant load.

Considering the latter case, the initial stress is the ratio of applied force in tension to cross-sectional area of the specimen. However, as elongation takes place, it is accompanied by a reduction in cross-sectional area, which increases the stress without having made any change in the force in tension.

For cylindrical specimens, not only is there a similar increase in stress resulting from reduction in cross-sectional area, but there is a further increase in stress due to the increased diameter as the specimen expands. Using the conventional hoop-stress formula, $S = PD/2t$ for the purpose of illustration, it is evident that for a constant pressure P, the stress S will increase directly as the diameter D, in addition to increasing inversely as the thickness t.

This analysis applies to perfectly concentric cylindrical specimens, which are not obtained in commercial production. The problem is further complicated by the factor of eccentricity. Since the stress varies inversely as the wall thickness, the initial stress in an eccentric specimen will vary around the circumference. The greatest strain and resulting reduction in thickness will occur at the thinnest part and the increase in stress will accordingly be greatest there, thus accentuating the effect of the eccentricity. Furthermore, as eccentricity of sheathing is generally non-uniform, the rate of increase of stress will be different for specimens not having identical cross-section. This places a definite limitation on the testing of cylindrical samples, in that reproducible results can be expected only from identical samples.

An attempt was made to broaden the field of reproducibility to include any degree of eccentricity by basing the test on constant stress instead of constant pressure. This required rather elaborate calculations and sliding scales involving the four factors in the equation for stress, as well as considerable dexterity on the part of the operator to keep pace with the changing conditions. This method was abandoned as too complicated, and the limitation accepted with respect to constant pressure and initial stress based on minimum thickness.

The decision as to the most advantageous length of test specimen was also influenced by the factor of eccentricity. The same factory limitations that make it impractical to manufacture perfectly concentric lead also operate to defeat the hope of obtaining long samples of the same degree of eccentricity. If the eccentricity changes in any given specimen, especially with respect to the minimum thickness, the stress will be changed longitudinally, and the time-to-burst may not be a function of the inside diameter and thickness as necessarily measured at the ends. Accordingly it was considered advisable to use specimens as short as possible that would leave a free length in the middle not restrained by the end clamps. Tests on samples of various lengths indicated that a 12-in. length is satisfactory for specimens up to 3 in. in diameter where the total expansion does not exceed approximately 25 per cent.

Reproducibility of Results:

In the latter part of 1935, results obtained by the authors were consistent enough to warrant a study of the reproducibility of test results in different laboratories. Accordingly, a cooperative program was arranged between the Brooklyn Edison Co. and the Philadelphia Electric Co.

Four 12-ft. samples of lead pipe were supplied by cable manufacturers, comprising one sample each of A.S.T.M. grade III and copper-bearing lead from the non-critical and critical regions. Half of each sample was sent to each laboratory.

The methods of conducting tests differed in the two laboratories in respect to sealing ends and applying pressure. Essential duplication was in the method of calculating stress and in holding the applied pressure constant. The schedule called for the testing of three specimens of each sample in each laboratory at the same stress, and the remaining samples at other values of stress.

The results of these tests were very promising. In the early part of 1937 a report was prepared and distributed to interested manufacturers and utilities, containing the following principal conclusions:

1. Constant-pressure hydraulic bursting tests of cylindrical specimens are reproducible among different laboratories.

2. A definite relationship exists between initial stress and time-to-burst.

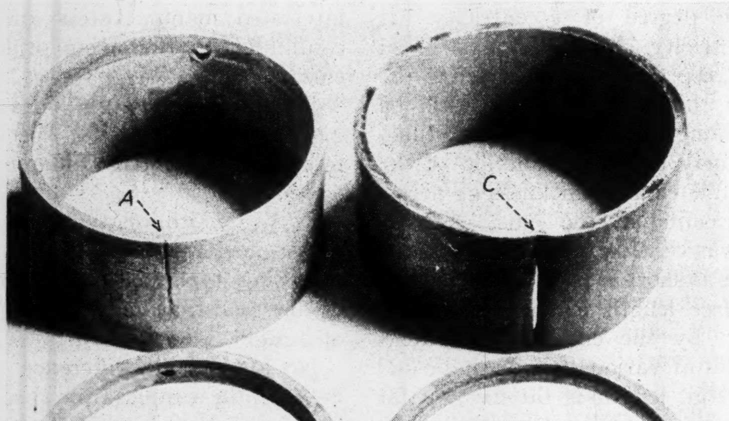
3. This type of test appears capable of distinguishing between certain types of lead.

No appreciable difference was found between the samples from the noncritical and critical regions. The relation indicated between initial stress and time-to-burst was that of a straight line when plotted on semilogarithmic paper, with a linear scale of stress as abscissas and a logarithmic scale of time-to-burst as ordinates. The line thus plotted for copper-bearing lead was approximately parallel to and definitely higher on the scale of time-to-burst than the lines for grade III lead. This might seem to confirm the conclusion of other investigators based on their tests that copper-bearing lead has superior characteristics for cable sheathing. It will be seen however that further developments leave this point open to question.

When the above report was issued no bursts had occurred at stresses below 800 psi. (95-lb. gage), but tests were in progress at 600 and 400 psi. (70 and 47-lb. gage). When failures occurred at these stresses it was found that the copper-bearing lead continued the straight-line relation between initial stress and time-to-burst to a value of 600 psi. For grade III lead, a knee apparently occurred at approximately

800 psi., and the line assumed a steeper slope in the direction of longer time-to-burst to a value of 400 psi. The lines for grade III lead and copper-bearing lead therefore converged, presenting the possibility that at normal operating stresses there would be little distinction.

The relation between initial stress and time-to-burst showed promise of pointing to a correlation between short-time and long-time tests. In the Brooklyn-Philadelphia project, the longest time-to-burst was approximately 1.9 yr. for a stress of 400 psi. at room temperature. It appeared desirable to seek even more consistent results so that extrapolations could be made to the period of normal life expectancy of


A
C
B
D
A & B-Same Failure Section in Adjacent Rings.
C & D-Same Failure Section in Adjacent Rings
Photomicrograph at A
Photomicrograph at D

Radial burst in die weld at 47-lb. gage pressure (max. initial stress 400 psi.); time to burst, 19 months.

Neck-down burst in lead between die and charge welds at 92.5-lb. gage pressure (max. initial stress 795 psi.); time to burst, 23 hr.

FIG. 6.—Hydrostatic Bursting Tests of Cylindrical Lead Specimens at Constant Pressure. Comparison of long-time and short-time bursts on two specimens from same sample.

cables. If this were possible, the relative advantages of pure lead and the alloys in common use could be determined.

Failures in Welds:

Up to this time little attention had been paid to the physical nature of bursts occurring under hydraulic pressure. In searching for possible causes for deviations from the indicated straight-line relation, it was observed that the nature of the burst was different for short and long-time tests. For short-time tests, bursts were of the wedge or knife-edge type previously described in connection with the bending test. As the value of stress decreased, and the time-to-burst increased, a point was reached where necking down of the lead preceding a wedge-type burst no longer occurred, and the sheathing failed abruptly with a square-edge fracture. These two types of failure are shown in Fig. 6.

It was at first assumed that these were breaks of the type best described as "intercrystalline," and previously reported by the I.P.C.E.A. Committee (8), Moore (11), and Phillips (12). The authors later observed that these bursts closely resembled the radial and rounded types obtained in the bending tests. Etching tests were then made of the long-time burst specimens and it was found that in every case so-called square-edge bursts had occurred in die welds or flow lines.

This was an unexpected development, because advancements in the cable industry since 1930 had been such that "cold welds" and "laminated lead" had been practically eliminated. The samples for the Brooklyn-Philadelphia project were of 1935 manufacture and contained welds that were considered as sound as the industry could produce at that time. The logical conclusion was that the previously accepted theory that good welds and flow lines were as strong or stronger than the associated lead did not apply under conditions of long-time stress. This revealed the probability that split sheathings that had been occurring in service were not necessarily confined to what had previously been termed defective sheathing.

Another factor involved in this problem was the fact that die welds were invariably located in the thinnest section of the cable sheathing. In addition, the point where the flow lines looped most directly from the inside to the outside of the sheath came in close proximity to the die welds. This gave rise to the condition of the greatest stress being applied at the weakest section of the sheathing.

Additional tests were made on sheathing of A.S.T.M. grade III lead, copper-bearing lead (0.06 per cent copper) and tin-bearing lead (2 per cent tin). In all cases, weld or flow-line failures were obtained by sufficiently reducing the stress. It was found that tin-bearing lead pipe required a longer time-to-burst at high stresses and a shorter time-to-burst at low stresses, as compared with copper-bearing lead. It was also found that tin-bearing lead sheathing failed in welds at higher stresses than copper-bearing lead sheathing, which in turn failed in welds at higher stresses than grade III lead sheathing.

A report on these findings entitled "Relative Strength of Welds of Extruded Lead Sheath or Pipe for Short-Time and Long-Time Bursting Tests" was presented at the November, 1938, meeting of the Technical Subcommittee of the High-Tension Cable Committee of the Association of Edison Illuminating Companies. The essential data are reproduced here as Figs. 6 and 7, together with the following summary and conclusions:

1. Lead pipe or sheathing which apparently has "properly joined" welds will probably fail in a weld in a long-time test at a relatively low stress, if the weld is present in the thinnest section.

2. Long-time tests on lead pipe or sheathing with welds not in the thinnest sections may produce different results from tests with welds in the thinnest sections.

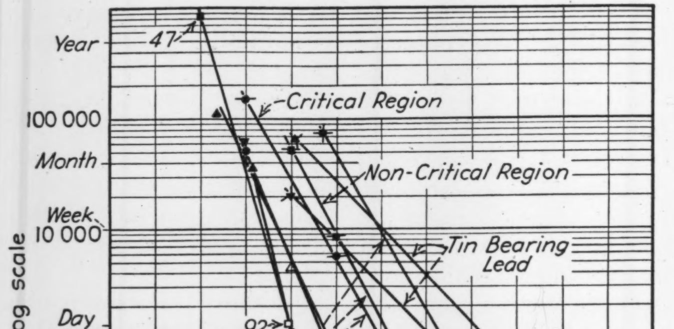


FIG. 7.—Showing Relation of Time-to-Burst *versus* Initial Stress.

3. Short-time bursting tests on lead pipe or sheathing including welds are not indicative of results to be expected in long-time bursting tests.

4. The etching test may be considered a criterion of proper joining or bonding of welds, but is unsatisfactory as a criterion of performance of pipe or sheathing under stress.

5. Pressure bursts which develop in service may occur in welds of cable sheathing that are either properly or improperly joined.

Cable manufacturers were reluctant to accept the theory that properly joined welds were not so strong as the metal itself. It was pointed out that the tests reported were not made on samples manufactured with the most modern presses, and that the latest improvements in extrusion technique

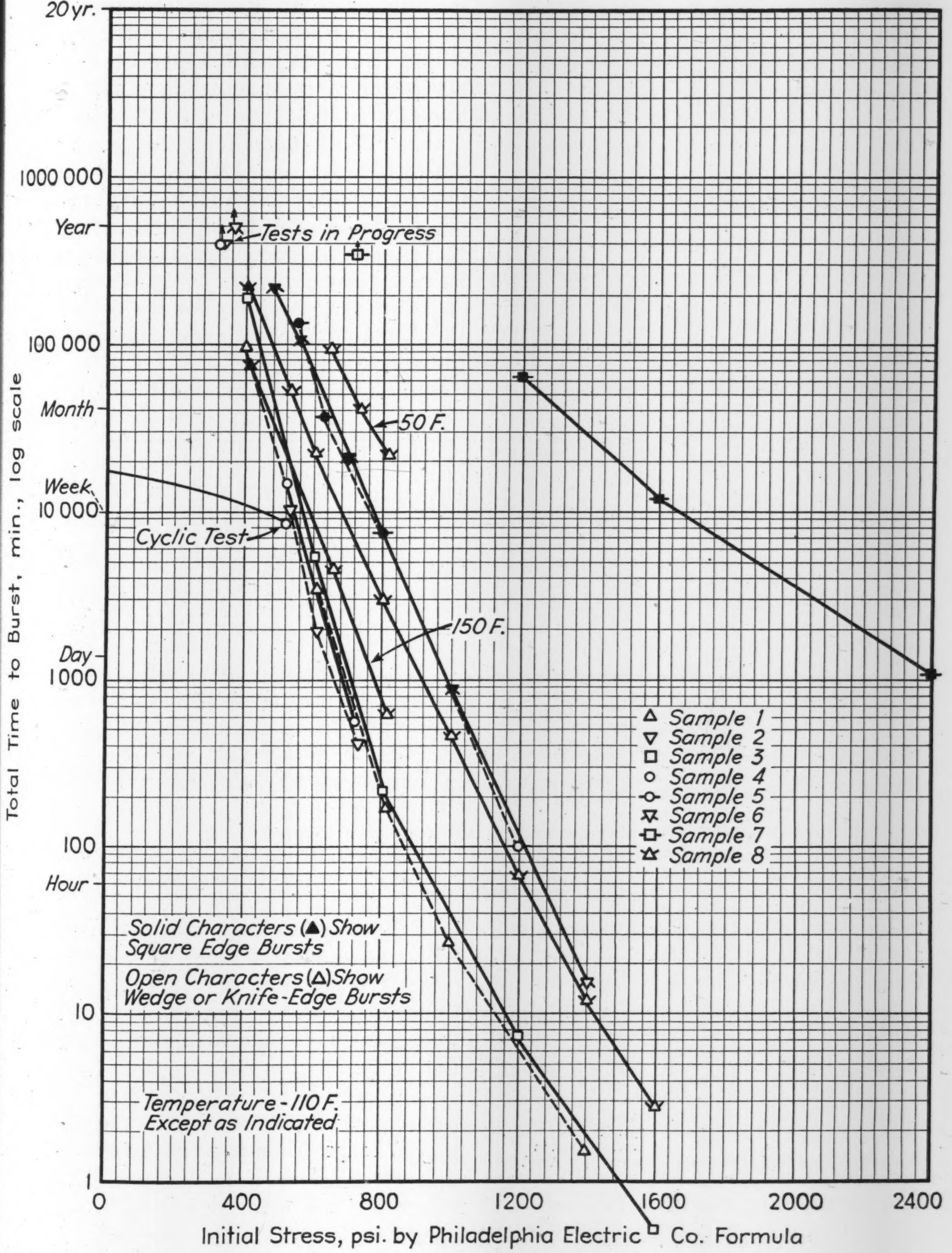


FIG. 8.—Showing Relation of Time-to-Burst *versus* Initial Stress.

might nullify the results. Consequently, it was decided to conduct additional tests—made at constant temperature—on lead pipe extruded with current manufacturing facilities.

Tests at a Constant Temperature of 110 F.:

The test results reported on samples listed in Table I are typical of those obtained from samples of pipe supplied by various manufacturers. All samples were approximately 2.75 in. in diameter and $\frac{9}{64}$ in. thick.

The relation of initial stress and time-to-burst for tests on these samples completed to date is shown in Fig. 8. Curves showing rate of stretch for all samples except sample 7 of calcium lead sheathing pipe are plotted in Figs. 9 to 12. In the case of sample 7 the total stretch was less than 1 per cent and satisfactory stretch data were not obtained.

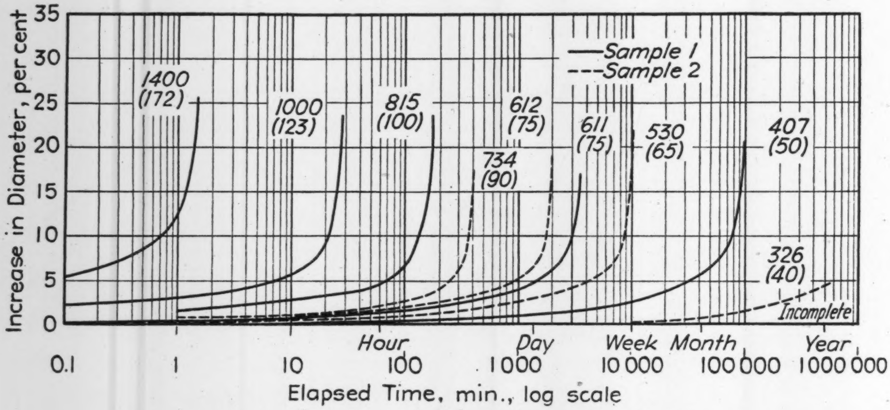


FIG. 9.—Showing Increase in Diameter *versus* Elapsed Time.

Cable sheathing pipe, special A.S.T.M. Grade III lead, noncritical region.

Reference numbers show initial stress in pounds per square inch by Philadelphia Electric Co. formula. Values in parentheses are corresponding constant gage pressures in pounds per square inch. Temperature, 110 F.

TABLE I.—IDENTIFICATION OF TEST SAMPLES.

Sample	Kind of Pipe	Manufacturer and Year	Kind of Lead
1 and 2[a]	Sheathing	A—1939	Special A.S.T.M. grade III noncritical region
3 and 4[a]	Sheathing	A—1939	Special A.S.T.M. grade III critical region
5	Sheathing	B—1938	Copper bearing
6	Sheathing	B—1938	Copper bearing
7	Sheathing	C—1938	Asarco
8	Slug press	D—1939	Copper bearing

[a] Sections of samples 2 and 4 were sent to H. F. Moore of the University of Illinois for cooperative creep tests.

It is not the purpose of this paper to draw definite conclusions from the limited data now available from these tests, but many possibilities present themselves as food for thought and more intensive research. It is considered sufficient for the present to submit the available data and point out some of these possibilities.

Samples 1 to 4, of grade III lead, were manufactured at the same time and in so far as is known differ only in their original position in the extruded pipe. Considering these samples as a group, when plotted semilogarithmically as in Fig. 8 the relation between initial stress and time-to-burst is that of a curve bending upward to the left, in the direction of longer time-to-burst for decreasing stress. This is in agreement with theoretical considerations from which it would be assumed that the curve becomes asymptotic to some critical value of stress requiring infinite time-to-burst.

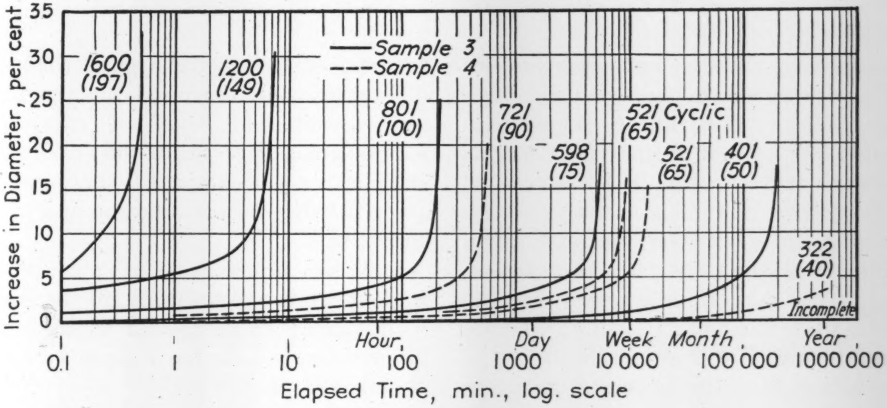


FIG. 10.—Showing Increase in Diameter *versus* Elapsed Time.*
Cable sheathing pipe, special A.S.T.M. Grade III lead, critical region

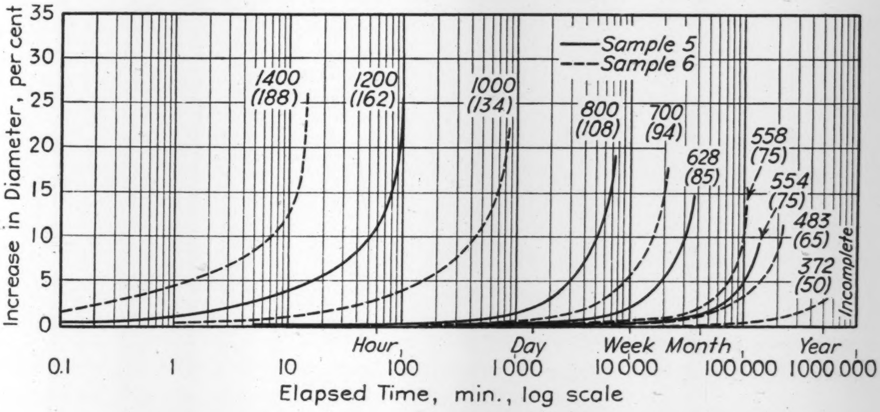


FIG. 11.—Showing Increase in Diameter *versus* Elapsed Time.*
Cable sheathing pipe, copper-bearing lead.

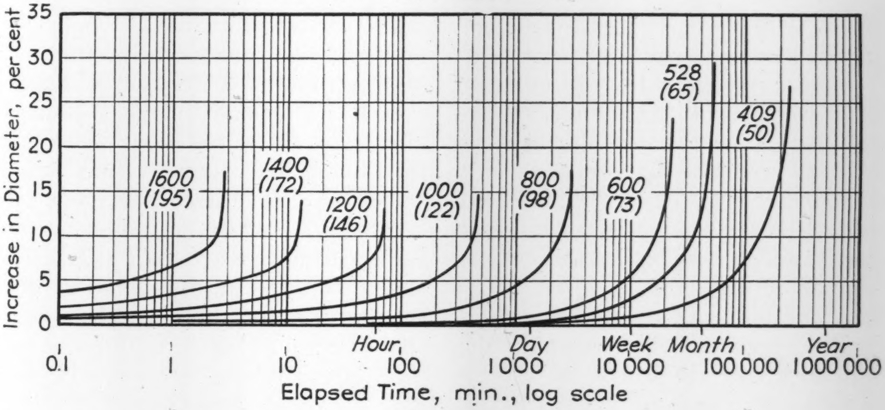


FIG. 12.—Showing Increase in Diameter *versus* Elapsed Time.*
Slug press pipe, copper-bearing lead, Sample 8.

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* Reference numbers show initial stress in pounds per square inch by Philadelphia Electric Co. formula. Values in parentheses are corresponding constant gage pressures in pounds per square inch. Temperature, 110 F.

Considering these four samples individually, it will be seen that although they fall in a very close group, each sample follows its own individual curve. Thus the curve for sample 1 is separated from that of sample 2, although both samples are taken from the noncritical region of the same pipe. The same is true of the curves for samples 3 and 4 taken from the critical region. It appears to be of special significance that the curves for samples 1 and 4 are in closer agreement than either of the pairs. It seems logical to conclude from this that the critical region is not a zone of weakness for the range of stresses between 1600 and 400 psi. (approximately 200 to 50-lb. gage).

TABLE II.—ANALYSES OF TEST SAMPLES.
Made by different laboratories as indicated.

Sample	Silver, per cent	Copper, per cent	Bismuth, per cent	Arsenic, Antimony, Tin, per cent	Iron, per cent	Zinc, per cent	Calcium, per cent	Cadmium, per cent	Nickel, per cent	Lead, difference, per cent	Laboratory
1.....	[a]	0.0001	0.15	....	....	....	....	....	....	....	E
2.....	[a]	0.0001	0.12	....	....	....	....	....	....	....	E
3.....	[a]	0.0001	0.15	....	....	....	....	....	....	....	E
4.....	[a]	0.0001	0.13	....	....	....	....	....	....	....	E
5.....	0.005	0.032	0.002	0.007	0.001	0.001	....	....	....	99.952	F
6.....	0.010	0.052	0.003	0.009	0.001	0.001	....	....	....	99.924	F
7.....	......	0.005	0.019	0.262	....	....	0.025	....	....	99.658	G
8.....	0.0046	0.0628	0.0578	0.0006	....	....	....	0.001	0.002	99.8712	H

[a] Not more than 1 oz. per ton (0.003 per cent).

NOTE.—Sample 5 is arbitrarily referred to in this paper as "copper-bearing lead" although it does not meet the requirements of the Specifications for Impregnated Paper Insulated, Lead Covered Cable, "Solid" Type, A.E.I.C. Sixth Edition, November, 1937, which states: "If copper bearing lead is specified, the copper content shall not be less than 0.040 per cent."

It is necessary to state the limits for this conclusion because all the bursts in this group were of the wedge or necked-down type not involving welds or flow lines. It is still to be determined whether weld failures will occur at lower stresses, and, if so, whether the critical region will be weaker.

With the influence of the critical region thus apparently eliminated, the reason for these differences in test results for samples so closely related is not obvious. This may be due to minor variations in the process of cable sheath extrusion. It may be argued that the differences noted are of no practical importance. This may be true, but it is of great importance that dissimilar samples are not compared when seeking a relation between short-time and long-time tests.

Samples 5 and 6 of copper-bearing lead sheathing pipe were of the same manufacture and in so far as is known differed only in their chemical content, as indicated in Table II.

It will be seen from Fig. 8 that the points for samples 5 and 6 could be plotted semilogarithmically as an identical straight line. This seems to indicate that, within limits, copper content does not affect time-to-burst. Although the points for sample 6 plot as nearly to a straight line as it is reasonable to expect of test results, the points for sample 5 do deviate from this line somewhat. If these deviations were accepted as reflecting the character of the sheathing rather than as irregularities of test results, the behavior of the sheathing would more nearly approach that of grade III lead; that is, the curve would tend to bend upward in the direction of longer time-to-burst for lower stresses. It may be significant that the curve for sample 5, which has less copper content than sample 6, behaves more nearly like those for grade III lead.

It is inconceivable that the points for lower stresses for samples 5 and 6 would continue the straight-line relation indicated in Fig. 8, because that would lead to the anomalous situation of obtaining a burst in a rather long time at zero stress. It becomes a point of interest then to learn where the knees in the lines for these two kinds of lead take place. It is of especial importance to determine whether and where the intersections take place for the curves for grade III lead pipe and copper-bearing lead pipe of various copper content. If these intersections occur at stresses above those existing in service, the superiority of grade III lead for cable sheathing will be established.

The comparison of grade III and copper-bearing lead pipe made above applies only to fabricated cable sheathing and is not a comparison of the two kinds of lead. For samples 5 and 6, the specimens at stresses of 1400 and 1200 psi. (188 and 162-lb. gage) had wedge-type bursts. The specimen at a stress of 1000 psi. (134-lb. gage) failed in a weld or flow line in an advanced stage of necking-down. The remaining specimens failed in welds or flow lines in progressively decreasing stages of necking down. It follows that a distinction must be made between the metal and the fabricated sheathing. It also follows that creep data, or the results of other tests on sheathing which does not include welds, does not permit drawing satisfactory conclusions concerning cable sheathing.

For sample 8, which was copper-bearing slug press lead pipe free of welds or flow lines, bursts of the wedge type were obtained for stresses down to 528 psi. (65-lb. gage). At a stress of 409 psi. (50-lb. gage) a failure of the intercrystalline type was obtained, but only after the specimen had stretched to practically the equivalent of a wedge-type failure. It remains to be determined whether results of these tests on slug press pipe are representative of results to be expected from sheathing pipe not involving weld failures. However, it is of immediate interest that the points for sample 8 plotted in Fig. 8 tend to give an upward curve similar to, although less steep than, the curves for grade III lead.

There is as yet insufficient data on sample 7, of calcium-lead pipe to warrant conclusions other than that the slope of the curve is less steep than for the other kinds of pipe tested to date.

In considering data for rate of stretch, the limitations are imposed that comparisons of stretch data are confined to specimens of a single sample, except for the general character of curves of different samples. This follows from the previously discussed observation that samples that are identical in other respects may vary in eccentricity, and therefore in rate of total stretch.

It is interesting to note the difference in the total stretch of copper-bearing lead pipe, with and without the influence of weld or flow line failures. Figure 11 shows increase in diameter plotted against elapsed minutes for the two samples of copper-bearing lead sheathing pipe. Figure 12 gives the same data for the sample of copper-bearing lead slug press pipe.

In Fig. 11, when weld failures are involved, decreasing values of total stretch are shown for decreasing values of stress. In Fig. 12, when wedge type failures are involved, the values of total stretch decrease for stresses from 1600 to 1200 psi. (195 to 146-lb. gage) and thereafter increase for stresses to 528 psi. (65-lb. gage). The single failure not of the wedge type at 409 psi. (50-lb. gage) showed a decrease in total stretch.

The significance of these observations is that in Fig. 11 the drooping characteristic of total stretch in relation to decreasing stress cannot be attributed to the kind of lead but must be attributed to the failures of welds or flow lines. It follows that this type of failure reduced the time-to-burst by the length of additional time it would have required for a wedge or intercrystalline type of failure.

How much weld failures would reduce the life of a cable in service is purely a matter of speculation. It is alarming, however, to note this downward trend of total stretch at stresses approaching those obtaining in service. In Fig. 11 the last completed test at a stress of 483 psi., on this particular size of pipe, was performed at a gage pressure of 65 lb. Unfortunately there is very little data available concerning values of internal cable pressures in service, but in a chapter contributed to a paper by Moore (13), Mr. Herman Halperin of the Commonwealth Edision Co. reports an actual peak of 72 lb.; further stating that a value of 50 lb. is about the maximum to be expected. From these data it appears that the test conditions reported here do not differ greatly from those in the field.

Data for a limited number of tests on sample 8 at temperatures of 50 and 150 F. are plotted on Fig. 8. These show the tremendous influence that temperature has on time-to-burst.

A point of difference between the tests heretofore discussed and service conditions is that the tests have been made at constant pressure whereas field pressures follow a loading cycle and may be a vacuum for part of the day. It is logical to question whether constant pressure tests are representative of cyclic conditions.

A single cyclic test has been made on sample 4 to investigate this point. The method is described earlier in this paper. Two cycles per day were used, with a maximum pressure of 65 lb. for approximately 2 hr., followed by approximately 20-in. vacuum. To simulate the loading cycle, the rate of heating was adjusted so that pressure was built up in about 1 hr. at an approximately uniform rate. After the 2-hr. period at constant pressure, heating was discontinued and the reservoir permitted to cool, the pressure decreasing to zero gage in about $1\frac{1}{2}$ hr. and to maximum vacuum in approximately 4 hr.

In computing the time during which the specimen was under stress, for comparison with an identical sample under constant pressure, it would evidently be improper to consider only the periods at maximum pressure. The time-to-burst for this cyclic test is plotted in Fig. 8, where an attempt has been made to show by a point the time-to-burst as a summation of the periods during which the specimen was at maximum stress; and also by a plotted line for which any point represents the total time for which the stress was greater than the value indicated. The relation between increase in diameter and time at maximum pressure for the cyclic test has been included in Fig. 10 for comparison with the constant-pressure tests.

While only one test has been made with this method, the result is important because it substantiates the results of creep tests reported by Gooding (14) at the April 1938 meeting of the A.I.E.E. In these tests it was found that when sufficient time was allowed between cycles of internal pressure tests for annealing of the lead, the total stretch was greater than for continuous test of the same duration.

CONCLUSIONS

The authors feel that the results of tests at a constant temperature of 110 F. have demonstrated the validity of the conclusions previously stated.[3] At this stage of the investigation any further deductions from test data appear unwarranted. It is perhaps more pertinent to list some of the questions which it is hoped that continuing investigations will definitely answer, as follows:

1. Has improved technique in the manufacture of cable sheathing eliminated the probability of weld or flow-line failures in A.S.T.M. grade III lead cable sheathing?
2. Does cable sheathing made of grade III lead have a longer life than the various alloys in use or advocated, under conditions of stress that obtain in service?
3. Does the similarity between "radial" or "rounded" bursts in welds or flow lines and "intercrystalline" bursts in homogeneous metal provide a clue to the reason for weld failures?
4. To what extent does the addition of alloys, in quantities as small as those used in so-called copper bearing lead reduce the life of cable sheathing by weakening the weld regions?
5. Considering that the occurrence of weld failures at higher stresses indicates relative weakness of the weld, will minor variations in the manufacturing process cause less uniformity in sheathing with relatively weak welds than in sheathing with relatively strong welds?
6. What, if any, allowance must be made for the effects of cyclic variations in pressure when using the results of constant-pressure bursting tests as indicators of service life expectancy of cable sheathing?
7. How does the temperature at which the cable is operated affect the results?

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[3] See pp. 893 and 896.

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[See Joint Discussion, p. 930.—ED.]

BURSTING TESTS AND TENSION TESTS FOR LEAD CABLE SHEATHING

By H. F. Moore,[1] C. W. Dollins,[1] and W. J. Craig[1]

Synopsis

In cooperation with the Philadelphia Electric Co., comparative tests to fracture under long-time steady load were made (1) by subjecting full sections of lead cable sheathing to internal oil pressure, and (2) by testing tension test specimens cut with the axis in a circumferential direction from pieces of the same sheathing. Four sets of tests were made, and there was found a good agreement between the nominal stress developed in the bursting tests and the tensile strength developed in the tension tests. There was also a quite good agreement between the increase of diameter in the bursting tests and the elongation of the tension test specimens.

Correlation of Bursting Tests and Tension Tests:

It has become quite generally recognized that, for metals which show an appreciable creep under long-continued steady load, short-time tests give little reliable information, either as to the amount of creep to be expected or as to the resistance of the metal to cracking and ultimate fracture under long-continued steady load. The problems arising from creep are very important in connection with lead and lead alloys used for sheathing for electric cables, and considerable study of the creep properties of these metals has been made, mainly by means of tension tests of specimens.

A length of cable sheathing in service is frequently subjected to internal pressure from the oil inside. This pressure sets up circumferential tensile stresses in the sheathing and at the same time longitudinal (axial) tensile stresses half as great as the corresponding circumferential stresses. Actual long-time tests of sheathing under internal hydrostatic pressure are being reported at this annual meeting by Phelps, Gates and Kahn[2] of the Philadelphia Electric Co. These long-time pressure tests are much more difficult and costly to make than are simple long-time tension tests of specimens cut from the sheathing, and an attempt to correlate the results of the two kinds of tests seems decidedly worth while.

Cooperation with the Philadelphia Electric Co.:

A general investigation of the creep and fracture of lead and lead alloys used for electric power cable sheathing is in progress in the Talbot Materials Testing Laboratory at the University of Illinois.

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[2] See p. 885.

This investigation is sponsored by the Utilities Research Commission of Chicago and is of direct interest to the Commonwealth Edison Co. In connection with this investigation an arrangement was made with Mr. Howard S. Phelps, Engineer of the Special Investigation and Testing Division of the Philadelphia Electric Co., whereby the results of a series of long-time bursting tests of sheathing, carried on at Philadelphia, were to become available at the Talbot Laboratory, together with unstressed pieces of the same sheathing from which tension specimens could be cut and tested.

Material and Sheathing Tested:

The sheathing studied in the bursting tests described in the paper by Phelps, *et al.*, comprised two lots of each of two different metals: (1) a "standard" grade III lead, and (2) a "special" grade III lead. Chemical analyses of these metals are given in the paper referred to. The outside diameter of the sheathing ranged from 2.83 to 2.88 in. The nominal thickness of wall was $\frac{9}{64}$ in.

Two test sections were cut from each of the pieces of sheathing. One test section for each metal was made from that part of the length of sheathing where the junction of two charges of lead was located. This was called the "critical" section. The other test section for each metal did not contain any "junction" metal, and it was called the "non-critical" section.

The details of the specimens for the bursting tests are shown in the Phelps-Gates-Kahn paper. The test specimens were full-section lengths of sheathing, 12 in. long, plugged at both ends, and kept during the test at a temperature of 110 F. In each bursting test specimen there was a circumferential tensile stress across the longitudinal weld in the sheath, and an axial tensile stress (half as great as the circumferential) along the weld. In each bursting test specimen from a "critical" section there was a circumferential charge weld.

Tension Test Specimens and Test Rack:

The tension test specimens whose strength and ductility were to be compared with the strength and ductility of the bursting test specimens were all cut from pieces of sheathing sent from the Philadelphia Electric Co. to the Talbot Laboratory. Each tension test specimen was cut with its axis in a circumferential direction, and each specimen reported in this paper contained a weld across its reduced section. It was, of course, necessary to flatten these specimens before making tension tests.

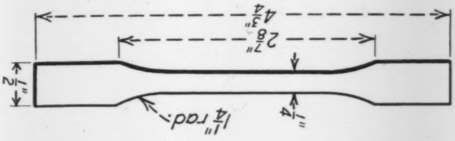

FIG. 1.—Tension Test Specimen. Nominal thickness $\frac{9}{64}$ in.

The location of the longitudinal weld was marked at Philadelphia on each piece of sheathing sent to the Talbot Laboratory.

Figure 1 shows the tension test specimen used, and Fig. 2 shows a test rack with the specimens enclosed in an insulated box fitted with double glass windows and heated electrically. The specimens were loaded by means of dead weights, and the temperature was held at 110 F. by means of a Fenwall temperature regulator.

Tensile Strength Under Long-Time Steady Load:

Figure 3 shows the tensile strength developed in the tension tests of specimens (solid circles) and also in the bursting tests (open circles). For com-

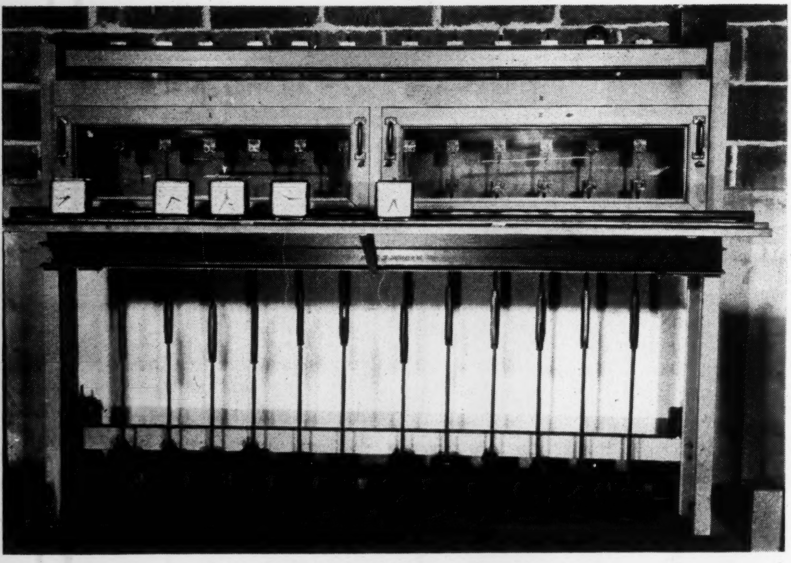

FIG. 2.—Test Rack for Tension Tests.

During the first week of a test the weights which load a specimen are attached by a cord to a trigger which when pulled stops a clock. If a specimen breaks, then the weights fall and pull the trigger. After a week, a twice-a-day inspection locates the time of fracture with sufficient accuracy.

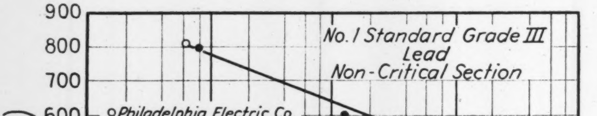

FIG. 3.—Tensile Strength - Time Diagrams for Bursting Tests and Tension Tests; Temperature, 110 F.

parison with the tensile strength of specimens, the tensile strength for bursting tests was computed by the thin-cylinder formula:

$$S = \frac{PD}{2t}$$

where

S = the tensile stress under a given internal pressure P in pounds per square inch,

D = the inside diameter of the sheath in inches, and

t = the thickness of the wall of the sheath in inches.

It will be seen from Fig. 3 that the time-strength diagrams for the bursting tests and for the tension tests are very nearly the same—so nearly the same that only one line is needed to represent both.

The apparent tendency of the time-strength diagrams for the tension test results for specimens of special grade III lead to become horizontal as time for fracture is increased is noted, and test results for bursting tests under several thousand hours are awaited with interest.

Elongation After Fracture for Bursting Tests and for Tension Tests:

Elongation after fracture and reduction of area are usually considered as measures of "static" ductility. The reduction of area cannot readily be measured in a bursting test, and to get a significant comparison of elongations it is necessary to find some base line for measuring elongation of tension test specimen which will give a significant comparison with the increase of diameter noted in the bursting test.

The outside diameter of the bursting test specimens averaged about 2.84 in., and this may be considered the gage line along which increase of diameter was measured. From Fig. 1 it is seen that the distance between shoulders of the unstressed tension test specimen is 2.875 in. Hence, a fair comparison of static ductility would seem to be the comparison of the percentage increase in diameter of the bursting test specimens with the percentage elongation between shoulders of the tension test specimens, since the gage lines for

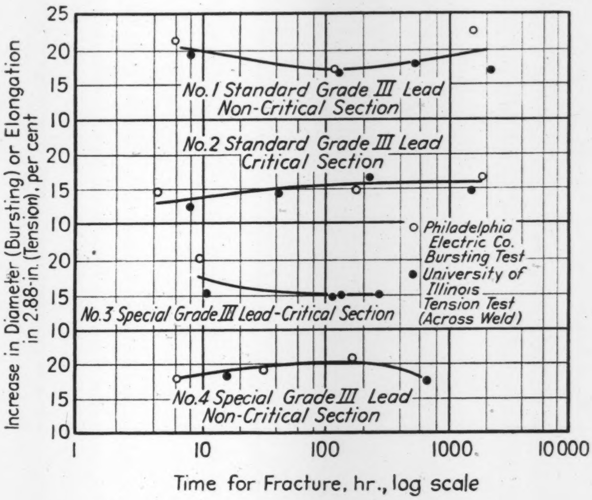

FIG. 4.—Elongation After Fracture for Bursting Tests and Tension Tests; Temperature, 110 F.

measurement of elongation are very nearly the same length.

Figure 4 shows the results of the tests. The agreement between bursting test results and tension test results is not so good as for the tensile strength results shown in Fig. 3, but with the exception of tests for over 1000 hr. on specimens of standard grade III lead, non-critical section, and the 10-hr. tests for special grade III lead, critical section, the agreement is fairly close. The elongation for special grade III lead, non-critical section, shows a tendency to fall off for tests which last for more than 200 or 300 hr., but general conclusions for these metals should not be drawn until test results for failures after several thousand hours are available.

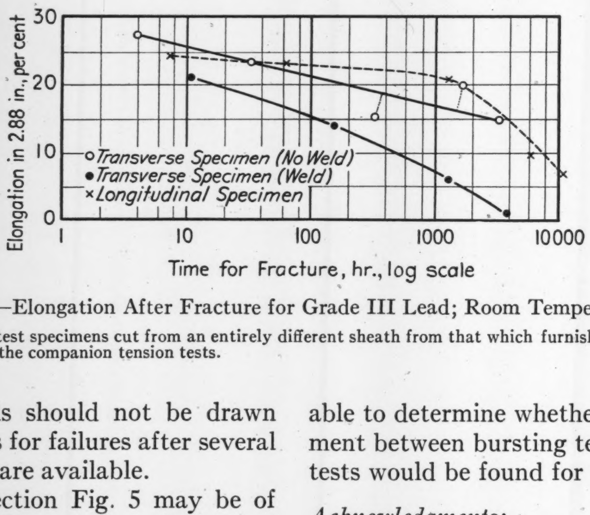

FIG. 5.—Elongation After Fracture for Grade III Lead; Room Temperature.

Results on tension test specimens cut from an entirely different sheath from that which furnished the specimens for the bursting tests and the companion tension tests.

In this connection Fig. 5 may be of interest. That figure shows changes of elongation with time required for fracture for tension test specimens of another lot of grade III lead, tested in the Talbot Laboratory some years ago. For this lead, whether tested circumferentially or axially or across a weld, ductility decreases very distinctly as the time for fracture increases, and in two of the three curves shown the elongation has reached very low values for tests extending to several thousand hours. Decrease of static ductility of lead and lead alloys with time under tensile stress seems to be a field well worth further investigation.

CONCLUSIONS

For the sheaths tested it seems that the prediction of tensile strength and static ductility under long-continued steady load could be judged as reliably from tension tests of specimens cut from sheathing as from bursting tests of short pieces of sheathing. Similar tests on sheathing made from other grades of lead and of sheathing made from some of the harder alloys of lead seem desirable to determine whether similar agreement between bursting tests and tension tests would be found for these metals.

Acknowledgments:

Acknowledgment is made to Howard S. Phelps of the Philadelphia Electric Co. for furnishing the material tested. Acknowledgment is made to the Utilities Research Commission of Chicago, which is sponsoring the investigation of lead sheathing now being carried on in the Talbot Laboratory at the University of Illinois. Especial acknowledgment is made to Herman Halperin, the chairman of the Advisory Committee of the Utilities Re-

search Commission for his helpful criticism of the testing and the preparation of this report.

The work at the University of Illinois is a part of the program of the Engineering Experiment Station, M. L. Enger, Director, and is a part of the research work of the Department of Theoretical and Applied Mechanics, of which F. B. Seely is the head.

[See Joint Discussion, p. 930.—ED.]

THE TESTING OF LEAD CABLE COVERINGS

By W. H. Bassett, Jr.,[1] and C. J. Snyder[1]

Synopsis

A method of making long-time burst tests is described as well as the various types of physical tests which have been made on lead cable coverings in order to correlate the quality of commercial material.

A new theory is suggested regarding the life of various extruded lead and lead alloy cable sheaths subjected to constant or intermittent tensile stress. The results indicate that at some low stress, such as 400 psi., all types of lead and lead alloys, regardless of their tensile strength as normally expressed, will fail in approximately the same period of time.

By making four bursting tests at different pressures the investigator can classify a given sample of lead sheath with respect to the softest lead or the hardest age-hardening alloy used by the power cable industry and can estimate its usefulness with the possible exception of resistance to corrosion and fatigue.

When metallurgists started to investigate the quality of lead-sheathed power cables in 1930, they found that split welds far overshadowed such other troubles as burst sheaths, wear at duct mouths, and crystallization of sheaths in manholes (**1**).[2] During the initial study, the cone expansion, the right angle bend, and the sheath stretching tests—originated to locate defective seam and charge welds—were of decided importance in evaluating the product of the cable sheath production units. With the general advent of better foundry practice at the lead presses, the defective welds were for all practical purposes eliminated and investigators turned their attention to the comparative life of lead sheaths under sustained loads.

Data collected from the hydraulic testing of lead cable sheaths over a period of years has shown that in all cases investigated lead and lead alloys lose their ductile wedge-shaped type of fracture as the pressure is decreased and the length of time to burst is increased.

In our early tests, we found that high-pressure, short-time burst tests always gave large increases in diameter with a sharp reduction in thickness at the fracture. Next, tests were used in which the pressure was gradually increased in steps, each pressure step being maintained for 5 or 10 min. In all of these accelerated types of tests, the final high pressure gave a sharp reduction of thickness at the fracture which obscured the brittle, blunt, intercrystalline failure pointed out by H. S. Phelps in 1936.

In our later tests, we have confined our burst tests to sustained pressures ranging from 500 to 2500 psi. hoop stress at room temperature on specimens selected from extruded pipe ranging from the softest lead to the hardest age-hardening lead alloy used in the power cable industry.

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[1] Manager, Metallurgical Development, and Engineer, Metallurgical Laboratory, respectively, Anaconda Wire and Cable Co., Hastings-on-Hudson, N. Y.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 929.

The samples selected for burst tests were subjected to various laboratory tests so as to have a complete case history of the samples and in order to make a definite comparison of test methods.

Chemical Analysis:

In Table I are given the chemical analyses of ten representative sample pipes. The presence of various metals was determined by spectroscopic examination and then the exact amount of the element was obtained by chemical determinations. The lead content was taken by difference. It is important that the composition be accurately known because the presence of certain impurities and common metals associated with lead influence the properties of the metal drastically, even when present in quantities as low as 0.01 per cent (**8**). However, the effect of many of these impurities on the service operation of lead sheaths has not been definitely established and since it has been shown that the addition of one impurity may counteract the harmful effect of another or even improve the finished product, much work remains to be done before a complete analysis specification for cable sheaths can be written.

TABLE I.—CHEMICAL ANALYSES OF BURST TEST SAMPLES.

Sample	Brinell Hardness Number	Lead, per cent	Bismuth, per cent	Copper, per cent	Antimony, per cent	Tin, per cent	Silver, per cent	Tellurium, per cent	Nickel, per cent	Calcium, per cent	Magnesium, per cent
No. 2264	3.9	99.920	0.065	0.013	0.0015	0					
No. 2261	3.9	99.921	0.066	0.011	0.0015	0					
No. 2180	4.5	99.880	0.059	0.059	0.002	0	0.0010[a]				
No. 2181		99.884	0.057	0.057	0.002	0					
No. 2747	(Similar to Samples Nos. 2180 and 2181)										
No. 2758	4.7	99.826	0.100	0.072	0.001	0	0.0015	0	0		
No. 2296	4.5	99.925	0.000	0.065	0.0003	0	0.005[a]		0.0044		
No. 2445	5.7	99.778	0.126	0.059	0.004	0	0.013	0.020			
No. 1228	10.4	99.905	0.000	0.053	0.0007	0	0.0062	0.000	0.0044	0.031	
No. 2856	13.1	99.658	0.019	0.005	0.002	0.26				0.025	0.031

[a] Nominal average.

TABLE II.—DIMENSIONS AND TENSILE PROPERTIES OF BURST TEST SAMPLES.

Sample	Outside Diameter, in.	Wall Thickness, in.		Tensile Strength, psi.[b]	Stretch, per cent[b]	Diameter Increase, per cent	Pressure Hoop Stress, psi.	Time, min.
		Avg.	Min.					
No. 2264	2.87	0.159	0.155	1905	46.6	42.8	1500	7.5
No. 2261	1.81	0.095	0.091	1823	57.7	25.7	1250	102
No. 2180	2.96	0.153	0.148	2220	44.5	30.5	1500	109
No. 2181	1.85	0.095	0.092	2136	44.5	27.0	1500	105
No. 2747[a]	2.95	0.146	0.131			8.5	1500	106
No. 2758	2.75	0.146	0.141			23.3	1500	67
No. 2296	2.75	0.139	0.132	2236	41.5	28.0	1500	103
No. 2445	2.81	0.137	0.130	2695	38.5	28.3	2250	17
No. 1228	2.35	0.117	0.109	4420	19.4	4.1	2500	3 800
No. 2856	2.71	0.140	0.135	5505	8.8	0.9	2500	12 500

[a] Sheath from cable.
[b] Ring-shaped specimens 0.75 in. wide used for test.

Tension Tests:

In Table II are reported the tensile strength and elongation of ten samples compared with the high-pressure, short-time burst test. It will be noted that when the internal pressure is such that the sample fails in approximately eight minutes, the percentage increase in diameter of the pipe compares favorably with the percentage elongation obtained in a standard tension test.

The authors have made numerous tension tests on lead sheaths, both by using a standardized shape of test specimen cut circumferentially and longitudinally from the lead tube and on carefully prepared rings of the material by stretching them over specially shaped end pins supported in the grips of a standard testing machine. A ring-shaped specimen is easier to prepare and has given very consistent results. It has the added advantages of not distorting the metal structure except during testing and of including in the gage length all the structural irregularities such as welds.

TABLE III.—DIAMETER CHANGES IN BURST TESTS AND BEND TEST RESULTS.

Sample	Short-time Series			Long-time Series			IPCEA Bend Test—2 lb. Tension —Right Angle Bends—Number		
	Diameter Increase, per cent	Pressure Hoop Stress, psi.	Time, min.	Diameter Increase, per cent	Pressure Hoop Stress, psi.	Time, min.	Avg.	Max.	Min.
No. 2264	42.8	1500	7.5	16.8	500	290 000	48	57	47
No. 2261	25.7	1250	102	13.8	600	101 000	65	75	55
No. 2180	30.5	1500	109	13.7	600	420 000	51	61	41
No. 2181	27.0	1500	105	20.5	800	52 000	51	55	45
No. 2747[a]	8.5	1500	106	8.0	800	83 000	48	53	27
No. 2758	23.3	1500	67	15.6	800	32 000	52	61	41
No. 2296	28.0	1500	103	14.3	800	47 000	45	52	37
No. 2445	28.3	2250	17	9.6	1500	40 000	39	45	33
No. 1228	4.1	2500	3 800	2.6	1500	55 000	17	21	15
No. 2856	0.9	2500	12 500	1.3	1500	153 000	25	35	21

[a] Sheath from cable.

The tensile strength of the lead has been found to be of little direct value but the elongation, or percentage stretch, is an important index of the ductility or plasticity of the cable sheath (**9**). It appears that a material showing less than 20 per cent stretch with a testing speed of 0.1 in. per min. per in. of gage length, at room temperature, may give difficulty when installing or removing a cable in the usual manner for underground service.

Draw-Bench Test:

It was not considered necessary to check the pipe samples with the draw-bench test as ring samples were used in making tension tests. This test was developed for mill or field use wherein the ring of lead is stretched over two specially shaped end pins held by the jaws of a portable draw bench. This test measures stretching qualities and shows up circumferential weaknesses such as defective radial welds (**10**).

Expansion or Drift Test:

Some of the ten samples were checked with the drift test to show that the pipe could be expanded satisfactorily at terminals or joints. This test also shows up circumferential weaknesses such as defective radial welds (**11**).

Bend Test:

In Table III are reported the average, maximum and minimum number of right angle bends obtained under 2-lb. tension when tested on an Insulated Power Cable Engineers Association type bend tester. In the bend test, narrow strips cut circumferentially from a lead sheath or tube are bent back and forth 180 deg. over a pair of standard radius jaws and the number of 90-deg. reverse bends to produce failure are counted (**1**). This test was originally intended to bring out lamination defects but is now being used to indicate nonuniformities in grain structure. An attempt has been made to evaluate lead sheath quality with the total number of bends. The variation in results caused by irregular grain size, the presence of normal circumferential charge welds, composition, and other minor details, make the test misleading and especially difficult to correlate with cable sheath performance.

Microstructure:

Microexaminations were used in a critical examination of the specimens after failure occurred in the burst tests (**12**).

Hardness:

The Brinell hardness values of samples have been included in Table I. It will be noted that in general the higher the lead content the lower the Brinell hardness value (**13**). Note the marked exception where calcium is present.

The authors have obtained very satisfactory and consistent results on material $\frac{1}{8}$ in. thick and over, using an Alpha Brinell machine fitted with a special aluminum weight arm which floats at 200 lb. (approximately 90 kg.). Hardness is an important test as it is an index of what may be expected in resistance to abrasion and scoring during shipment and installation of cables, as well as in resistance to cutting by support rings when installed overhead.

Fatigue Tests:

While fatigue tests have been conducted on some of the samples referred to in Tables I and II, the results are not included here since the subject cannot be properly treated in so short a space. There appear to be different types of fatigue phenomena for lead which may require several kinds of tests to indicate its fatigue resistance for different types of cable service (**3**, **14**). As an example, materials that are resistant to vibration or to rapid slight bending are not necessarily resistant to slow and more severe bending. The intercrystalline failures obtained by static loading, known as stress cracking, such as are experienced in creep and burst testing, have the same appearance as fatigue fractures and are considered by the authors in the same category as fatigue, although by definition they cannot be counted as such. Fatigue resistance is an extremely important property for many lead-covered power cable installations and this subject is receiving more attention.

Creep Tests:

Loading tests made by suspending weights from free-hanging strips of lead and by subjecting measured rings or tubes to internal hydrostatic pressure have shown some interesting results but they are still in the development stage and their practical importance has not yet been determined. Some creep data are reported in a later paragraph. There are two values to be considered in the practical application of creep test data: first, the initial set or deformation each time a stress is applied, and second, the rate of deformation caused by a sustained stress (**2**, **15**).

Burst Tests:

Tests were made of tube specimens of various sizes and lengths and sheath specimens removed from cables by sealing the ends and subjecting them to constant sustained internal pressures until failure occurs. Various pressures have been used so that the test time varied from a few minutes to several years.

By maintaining the internal pressure at a low, constant value, the time to burst was greatly increased and the percentage increase in diameter was altered so that the values simulated those obtained in service. It was also noted that the character of the fracture changed from a ductile necked-down separation to a brittle, blunt, intercrystalline failure. The tests of sheath from cable were the exception which did not follow this rule.

This marked change in character of the burst samples in the hydraulic tests was noted in not only the soft, pure lead specimens but also in all of the alloys tested, including the strongest age-hardening materials, which have been used for power-cable sheaths.

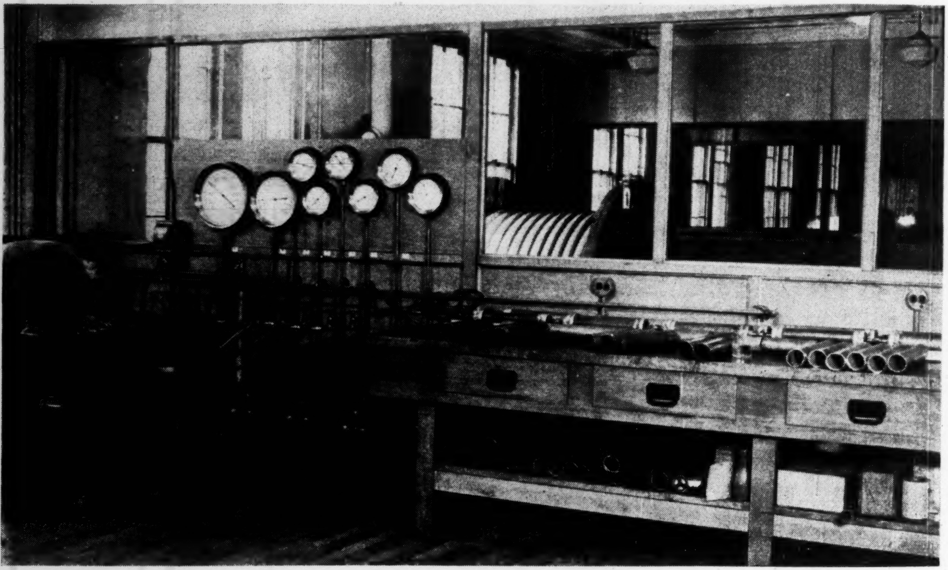

FIG. 1.—Apparatus Used for Making Constant Pressure Burst Tests on Lead Pipe.

A comparison of the tests made in our own as well as other research laboratories confirms these tendencies. Note in Table III the marked difference of percentage increase in diameter of specimens tested at high pressures failing in a few minutes compared with the result obtained at low pressures which required several thousand minutes to fracture.

The long-time, low-pressure variety, wherein the pressure used was about equal to the maximum obtained in service, was found especially desirable as it showed useful information regarding life and failure characteristics of different types of lead. This test also served for detecting structural weaknesses and for obtaining practical creep data as well as data on resistance to stress cracking under sustained loads. Although this test is still in the development stage its importance will be understood by a review of the authors' results. It shows by one set of tests the necessary engineering information except fatigue and corrosion resistance, for distinguishing between good and poor lead-sheathing materials.

EXPERIMENTAL DATA FROM BURST TESTS

Apparatus:

A part of the apparatus used for the burst tests is shown in Fig. 1. A motor-driven gear pump is connected to a reservoir and by-pass system to supply any desired internal pressure in each lead sample. The piping diagram is given in Fig. 2. The exact pressure is shown on

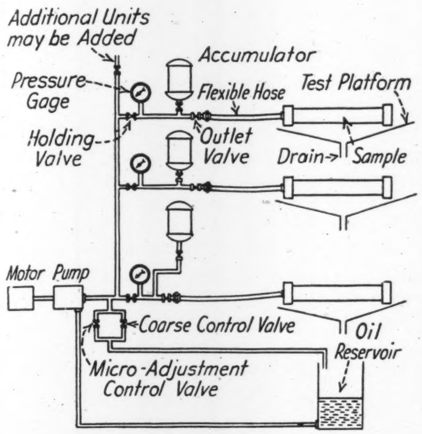
Additional Units may be Added
Accumulator
Pressure-Gage
Flexible Hose
Test Platform
Holding-Valve
Outlet Valve
Drain
Sample
Motor Pump
Coarse Control Valve
Oil Reservoir
Micro-Adjustment Control Valve

FIG. 2.—Schematic Diagram of Pressure System for Multiple Constant Pressure Burst Tests.

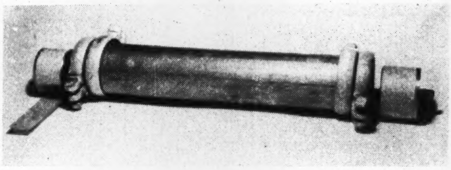

FIG. 3.—Lead Tube Specimen 15 in. Long Ready to Be Assembled for Burst Test.

the gages while the accumulators on the floor below hold the pressures constant over long periods of time. The design and layout of equipment for these tests is inexpensive but care must be taken to provide for accurate reading and adjustment of the individual pressures and for maintaining them constant.

The ends of short test specimens are tightly clamped with compression fittings similar to those originally suggested by Phelps and completely filled with oil or water. Long specimens tested at low pressures are filled with compressed air. The fittings now in use consist of smooth machined steel plugs approximately 0.020 in. less in diameter than the inside diameter of the tube. These are wound with two or more layers of ordinary gummed paper, slipped inside the end of the tube and then clamped tightly with two malleable iron hose clamps on each end as shown in Fig. 3. These fittings are easy to apply and remain tight.

Expansion Curves:

Many samples of lead and lead alloys were tested at various pressures and the amount of stretch recorded against total

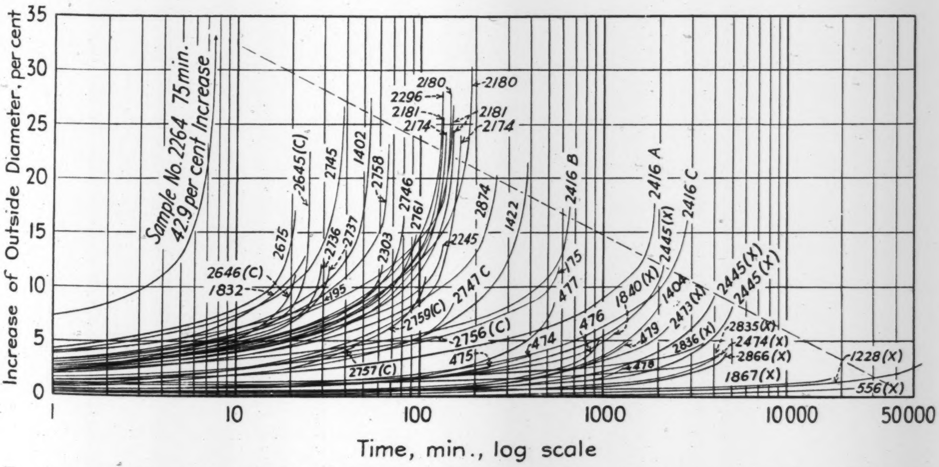

FIG 4.—Expansion Curves Obtained from Burst Tests at 1500 psi. Hoop Stress on Different Samples of Lead and Lead Alloys.

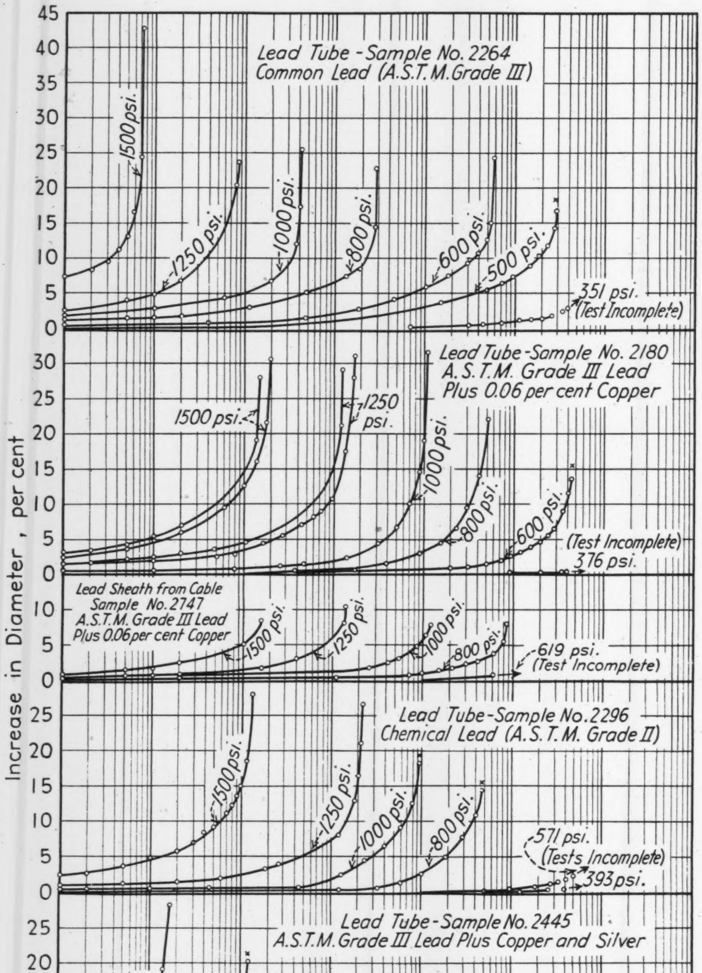

FIG. 5.—Expansion Curves Obtained from Burst Tests at Different Pressures on Lead Samples.

time under stress. The pressures were held constant well within ± 2 per cent for 6 days each week. A test at stress of 1500 psi. was found most useful as a preliminary test on an unknown sample to determine the characteristics of the material. Figure 4 shows the results of many tests at 1500 psi. stress. Some materials such as sample No. 2264, lasted only a few minutes whereas others, such as sample No. 1228, lasted over 50,000 min. (approximately one month) before failing. In general, the weaker materials which failed in the shortest time stretched more than the stronger ones. An average line has been drawn through the end points of these curves to indicate those materials which have better ductility than the usual run of their class. As pointed out previously ductility is an important property.

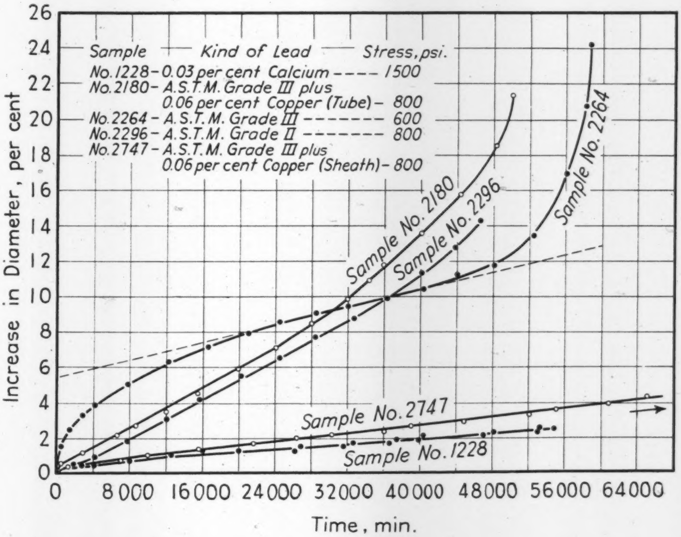

FIG. 6.—Expansion Curves Obtained from Burst Tests Plotted for Determination of Creep Rates.

On the other hand, when a curve falls far short of the average line it shows that the material is stiff, brittle, and unsuitable. When testing samples removed from cables instead of extruded as empty tubes, the amount of stretch by this test was found to be materially less and a different average line must be used. In Fig. 4, curves marked with a (*c*) after the number represent cable sheath samples. All these tests and others shown in subsequent figures were made at normal room temperatures.

After a sample had been given a preliminary test at 1500 psi. stress, additional tests at lower or higher stresses, as indicated by the result, were made to find the characteristics of the sample. Figure 5 shows, as examples, the results of six groups of these tests on six different samples. The internal pressure was held constant throughout each test. The increase in diameter has been plotted as ordinate against time as abscissa on semi-logarithmic paper. It will be noted that the specimens tested at the higher pressures stretched the most before failing.

The duplicate tests at 1500 and 1250 psi. for sample No. 2180 show what was obtained in the way of checks on different specimens selected at random and tested at different times.

Creep Data:

When burst test data are plotted on rectangular coordinates the tests which have a life of at least 30,000 min. can usually also be used for obtaining creep data. As an example, Fig. 6 shows five curves of this type plotted on a 60,000-min. scale. Creep rates for these tests were determined by extending the straight portions of these curves and are listed in Table IV. The initial set was taken as the difference between the origin and point of intersection of the index line and extended straight line.

TABLE IV.—CREEP RATES CALCULATED FROM LOW PRESSURE BURST TESTS 15 IN. LONG TUBE SAMPLES, ROOM TEMPERATURE.

Sample	Time Tested, hr.	Hoop Stress, psi.	Initial Set, per cent	Creep Rate, per cent per 1000 hr.	Total Creep, per cent in 1000 hr.
No. 2264	978	600	5.4	7.4	12.8
No. 2261	1681	600	4.4	5.55	9.95
No. 2180	851	800	0.40	16.84	17.24
No. 2180	7046	600	0.17	1.47	1.64
No. 2181	857	800	0.38	18.1	18.5
No. 2747	1380	800	0.44	3.46	3.90
No. 2296	777	800	−0.3	16.70	16.40
No. 1228	579	1750	0.77	6.03	6.80
No. 1228	912	1500	0.43	2.33	2.76

The composition and dimensions of all the samples referred to in Figs. 5 and 6 are shown in Tables I to III. All stresses were calculated by the formula $PD = 2\ St$, where D equals the inside diameter and t equals the minimum wall thickness and all samples tested were known to have a uniform grain structure.

A complete record of diameter, wall thickness variations, weld locations, and production data was kept, but since the specimens invariably failed in or near the thinnest area, these data are not given.

Life-Stress Curves:

When the data from these groups of burst tests are plotted in still a different way with the time for failure as ordinate on a logarithmic scale against the stress as abscissa, a group of curves shown in Fig. 7 was obtained. These curves show the relative life of a sample at any stress.

Most of the curves are straight lines indicating that the life of a lead tube bears a definite relationship to the stress imposed, and the life varies as an exponential power of the stress. The curves for samples Nos. 2264 and 2261, A.S.T.M. Grade III lead, show the lowest strength and the steepest slope. Between 800 and 1000 psi., all the curves for this grade of lead show a break upward, indicating that common lead becomes relatively stronger as compared with other materials as the stress is reduced. The curves for copper lead and chemical lead are usually straight lines with a uniform but flatter slope than common lead.

The curves for the lead alloy materials are also usually straight lines but have a still flatter slope which means a comparatively poorer life at low stresses. The curve for sample No. 2445 is drawn with a break to the left at the 2000 psi. ordinate thus indicating comparatively poor life at the lower stresses, and other samples of lead alloys have also given a similar shape of curve. As the stress is lowered, these alloy materials show a comparatively less rapid increase in their life as compared with lead, so that, for example, at a stress of 1500 psi. calcium lead would have an expected life of 6000 times that of common lead, but at a

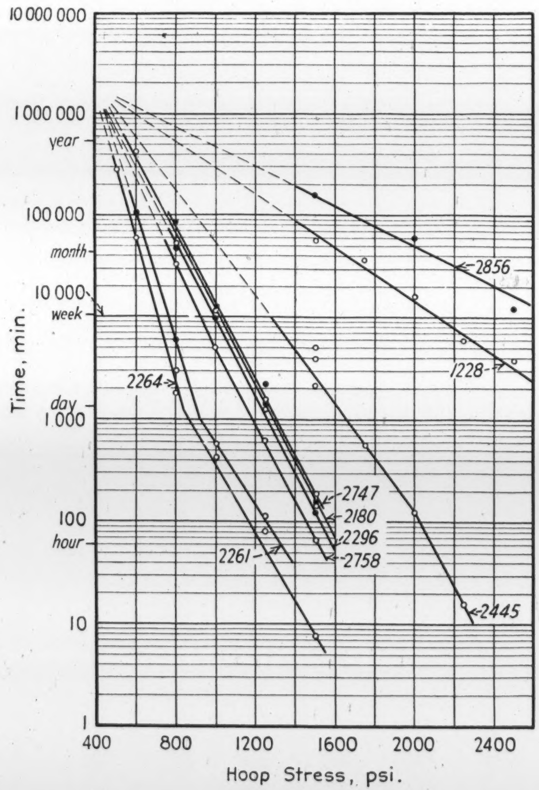

FIG. 7.—Summary of Authors' Data Obtained from Burst Tests on Lead Tubes of Various Compositions.

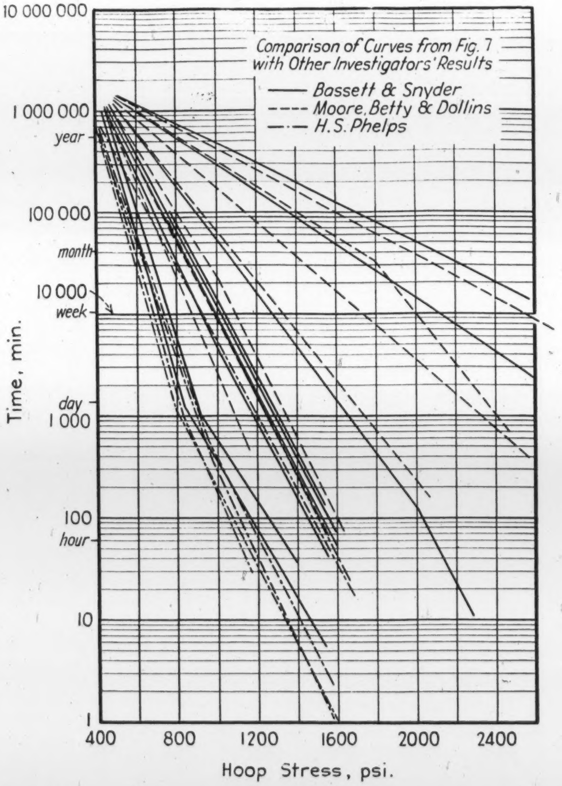

FIG. 8.—Comparison of All Life-Stress Curves, Obtained by Moore, Phelps and Bassett.

stress of 800 psi. calcium lead would have an expected life of about 100 times that of common lead.

Two special samples tested at 800 psi. stress at the same time under similar conditions lasted 66,000 min. for the common lead and 164,000 min. for the calcium lead, a ratio of only 3 to 1. Neither of these samples failed in a weld.

From the data recorded here it appears that at a stress of approximately 400 psi. nearly all the lead alloy materials would have about the same life of approximately 2,000,000 min. (approximately 4 yr.). Additional tests at 400, 500, and 600 psi. on all the samples mentioned in Fig. 7 are now running but some are incomplete. The authors have many more sets of tests which are not shown because their proximity to each other would make it difficult to read an individual curve. The data shown were obtained on samples produced on one lead press but the tube samples or sheath samples extruded by five manufacturers and obtained from different types of extrusion presses and on three different types of die blocks have also been tested. The variation in apparent life at 400 psi. stress of the various samples has been small, testing between 1,500,000 and 2,500,000 min., but a few especially prepared samples were exceptions.

The theory that nearly all lead and lead alloy materials will have about the same life when stressed at some relatively low stress is also indicated by data published by Moore and others (2 and 3). The authors have taken the liberty of redrawing in Fig. 8 some of Moore's curves (3) from his Fig. 25.

In a paper presented before a meeting of a committee of the Association of Edison Illuminating companies, Phelps (4) presented data and curves obtained by his constant pressure hydraulic bursting tests on lead cable sheath specimens. The authors have taken the liberty of redrawing his Fig. 1 in our Fig. 8. This figure shows the very close agreement between the results obtained by Moore and the authors and shows that Phelps' curves are similar but aim for a lower common point.

Type of Fracture:

Another detail of the burst tests on lead pipe is the appearance of the fracture when a specimen fails. It was found that all the soft lead No. 2264 specimens tested above 500 psi. necked down to a knife-edge when they failed, whereas none of the hard alloy specimens, No. 1228, necked down, but instead failed with square edged intercrystalline fractures.

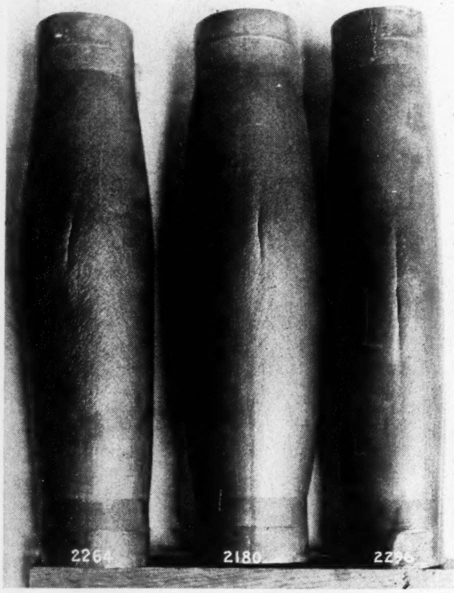

FIG. 9.—Appearance of Failed Specimens of Lead Tube Samples Tested at 1250 psi. Sample numbers indicated on specimens.

The type of failure on the other samples of intermediate hardness varied as shown on the curves in Fig. 5, the small

(x) at the end of the curves indicating an intercrystalline fracture. The ductile type of fracture obtained on three types of lead is shown in Fig. 9 tested at 1250 psi. This type of fracture is variously referred to as knife-edged or wedge-shaped fracture, necked-down separation, ductile tear, etc., and is characterized by practically 100 per cent reduction in area. The intercrystalline failures are characterized by practically no reduction in wall thickness at the edges and the separation, which may or may not be connected with a weld, follows the grain boundaries. It frequently happens, however, that one of the welds is located in the thinnest part of the tube wall and because it offers the shortest grain boundary path across the wall, the failure occurs at the weld. Figure 10 shows an intercrystalline failure in sample No. 2856 tested at 800 psi. stress. Evidence that these cracks are intercrystalline and not simple weld failures is shown in later figures but for the present it is pointed out by Table V that the tendency to intercrystalline failure bears a definite relationship to the hoop stress and ductility of the lead. It will also be noted that as the Brinell hardness increased a higher stress was required to obtain a ductile wedge-shaped fracture.

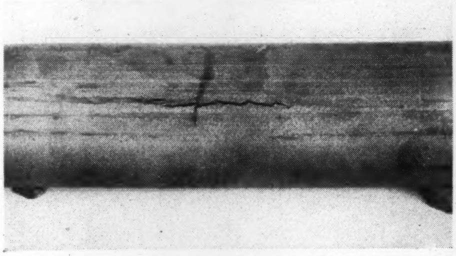

FIG. 10.—Typical Intercrystalline Type of Failure Produced by Low-Pressure Long-Time Burst Test. Sample No. 2856 tested at 800 psi. stress.

TABLE V.—TENDENCY TO INTERCRYSTALLINE CRACKING SHOWN BY BURST TEST SPECIMENS TESTED AT VARIOUS STRESSES.

Sample	Brinell Hardness Number	Stress, 2500 psi.	Stress, 2250 psi.	Stress, 2000 psi.	Stress, 1750 psi.	Stress, 1500 psi.	Stress, 1250 psi.	Stress, 1000 psi.	Stress, 800 psi.	Stress, 600 psi.	Stress, 500 psi.
No. 2264	3.9					—	—	—	—	—	x
No. 2261	3.9						—	—	—	x	
No. 2180	4.5					—	—	—	x	x	
No. 2747	—					—	—	—	—		
No. 2758	4.7					—	—	x	x		
No. 2296	4.5					—	—	x	x		
No. 2445	5.7		—	x	x	x			x		
No. 1228	10.4	?	x	x	x	x			x		
No. 2856	13.1	x		x		x					

— Ductile fracture.
x Intercrystalline fracture.

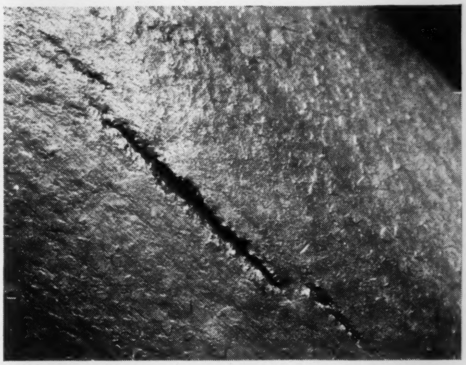

FIG. 11.—Surface Appearance of 600 psi. Specimen, Sample No. 2261, at Point of Failure Showing Grain Movement and Intercrystalline Cracks (× 4). Oblique lighting.

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When a lead is stressed at a sufficiently low stress so that little or no plastic flow occurs, it usually fails with an intercrystalline break (5). The practical value of the above observation remains to be discovered since we showed in Figs.

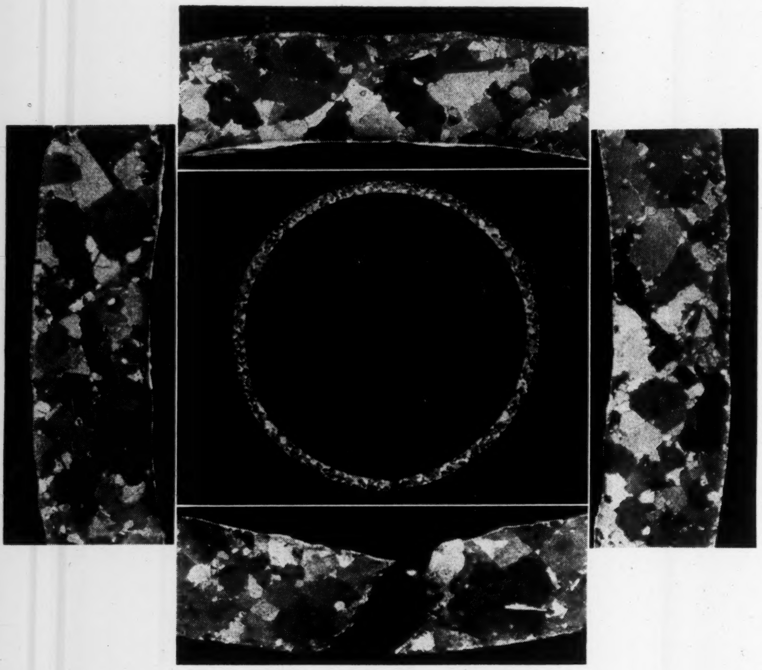

FIG. 12.—Structure of Cross-Section of 600 psi. Specimen of Sample No. 2261 After Test, Showing Type of Failure and General Intercrystalline Cracking. Ring (× 1.5). Micrographs (× 15).
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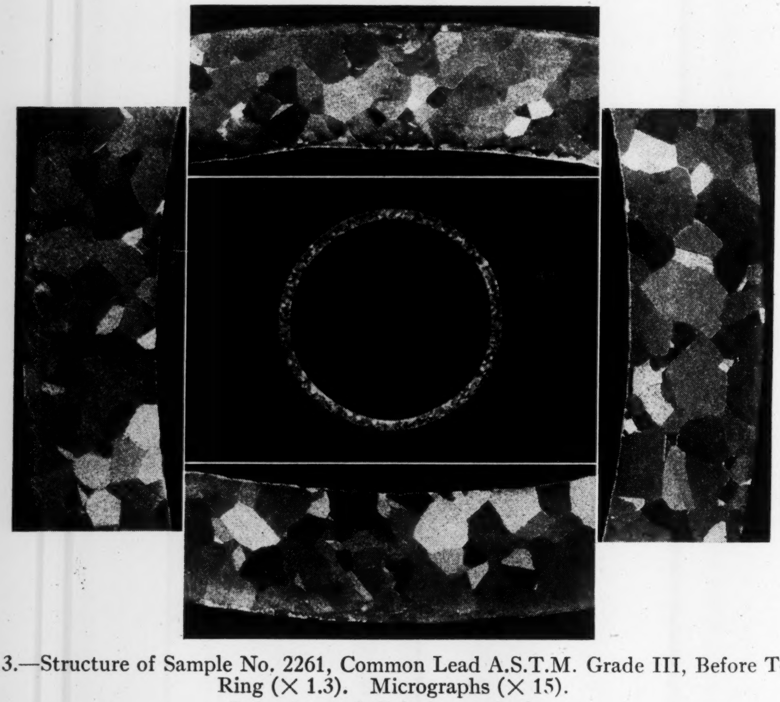

FIG. 13.—Structure of Sample No. 2261, Common Lead A.S.T.M. Grade III, Before Testing. Ring (× 1.3). Micrographs (× 15).
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7 and 8 that the life of a test specimen bore a definite relationship to the test stress regardless of the type of failure.

One practical benefit of this information may be a better understanding of the cause of certain service failures of lead cable sheaths. We note that Halperin (7) stated in a recent discussion before the American Institute of Electrical Engineers that after 5-yr. service of a certain 66-kv. cable using oil pressure joints, he was experiencing lead sheath failures in greater number than on other cables which had joints with no pressure applied. It is possible that the sheath failures on the cable using pressure joints were in some way a result of stress cracking.

There have been rare exceptions to both of these rules, however, as we have pointed out above and by investigating the exceptions something of value may be found. It seems logical that if intercrystalline failures could be averted, the burst samples should last longer than otherwise, although tests have not indicated such to be true.

Microstructures:

A microscopic investigation was made of various burst test specimens that had failed with intercrystalline fractures.

The 600 psi. specimen of common lead, sample No. 2261, showed indications of movement of the grains one upon the other without any general breaking up or refinement of the original grains. In Fig. 11 is shown the outside surface of the tube where it failed. The oblique lighting shows that the original grains have moved by apparent slippage or rotation of the grains one upon the other, to different elevations without any considerable rounding of the edges. It can also be seen that there are irregularities in the die scratch lines on the surface, which indicates that the grains have rotated in a horizontal plane as well as vertically (2).

The cross-sectional structure of the tube after test is shown in Fig. 12. The ring in the center is a complete section magnified 1.5 times and the side micrographs show the structure at 15 diameters of the areas to which they are tangently mounted. The bottom micrograph shows that the failure was strictly at grain boundaries and each of the other micrographs shows one or more intercrystalline cracks. There were many such cracks in the structure where failure could have occurred, indicating that the two radial welds in the tube were not necessarily a factor in the failure. The grain size and appearance was approximately the same after testing as it was before testing, the only difference being the formation of many small grains at the boundaries of the original large grains. Figure 13 shows the original structure of this tube.

Other specimens of common lead showed substantially the same structure. It therefore appears that the stress was insufficient to cause plastic flow of the metal but sufficient to cause the grains to rotate to a more favorable orientation thus setting up stress concentrations at certain grain boundaries which caused them to fail.

The 800 psi. specimen of copper lead, sample No. 2180, showed more surface cracks and more grain deformation than the common lead materials. Figure 14 shows the outside surface of the tube which has been lightly etched to show that the cracks are intercrystalline. The cross-sectional structure of the tube after test appears in Fig. 15. These micrographs show very prominent grain boundaries with many small cracks throughout the section. The general grain size was not changed by the test but the shape of the grains was changed from an irregular mass to a more or less

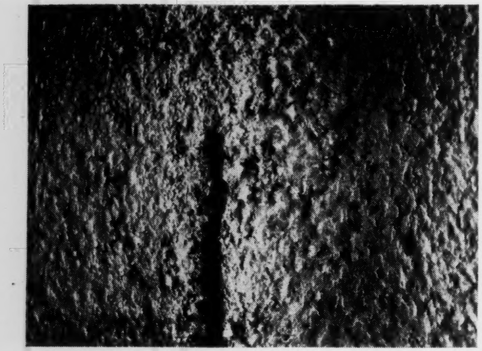

FIG. 14.—Surface Appearance of 800 psi. Specimen, Sample No. 2180, After Test, Showing General Intercrystalline Cracking Around Square-Edged Failure (× 4). Surface lightly etched.

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diamond pattern. There also appears to be a change in orientation of the grains as is shown by the change from the white and black shades to a more uniform dark gray shade when etched. Figure 16 shows the original structure of the sample. Figure 17 shows the structure at a magnification of 100 diameters to illustrate clearly the type of small intercrystalline cracks many of which were found throughout the section. The irregular curly grain boundaries after test are also a point of interest.

Most of the alloy lead specimens showed much more extensive intercrys-

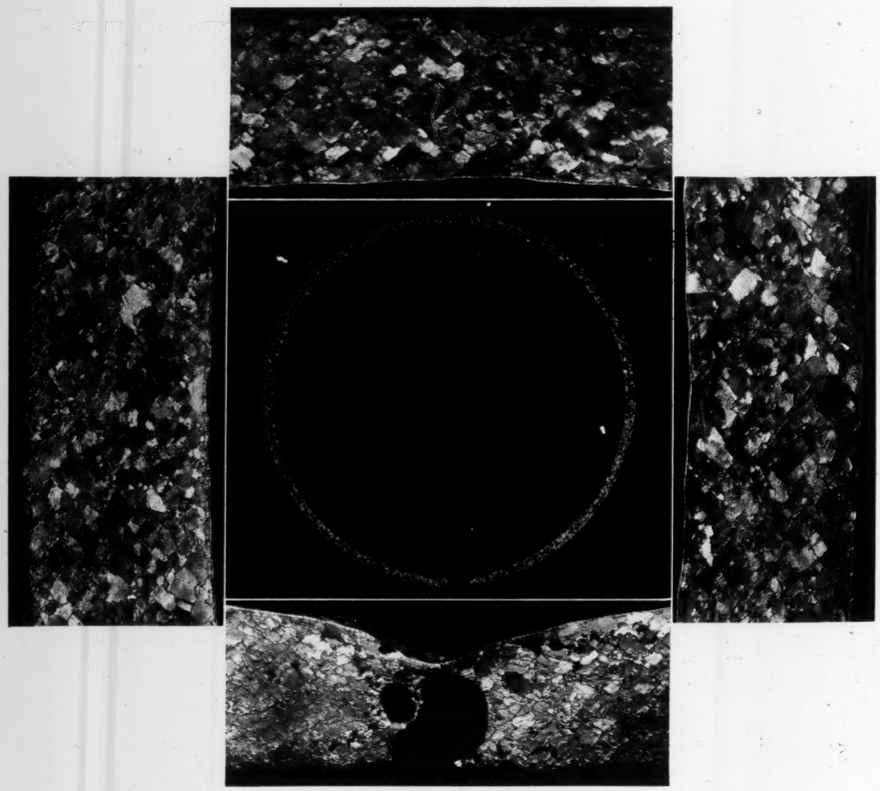

FIG. 15.—Structure of Cross-Section of 800 psi. Specimen of Sample No. 2180 After Test, Showing the Type of Failure and General Intercrystalline Cracking. Ring (× 1). Micrographs (× 15).

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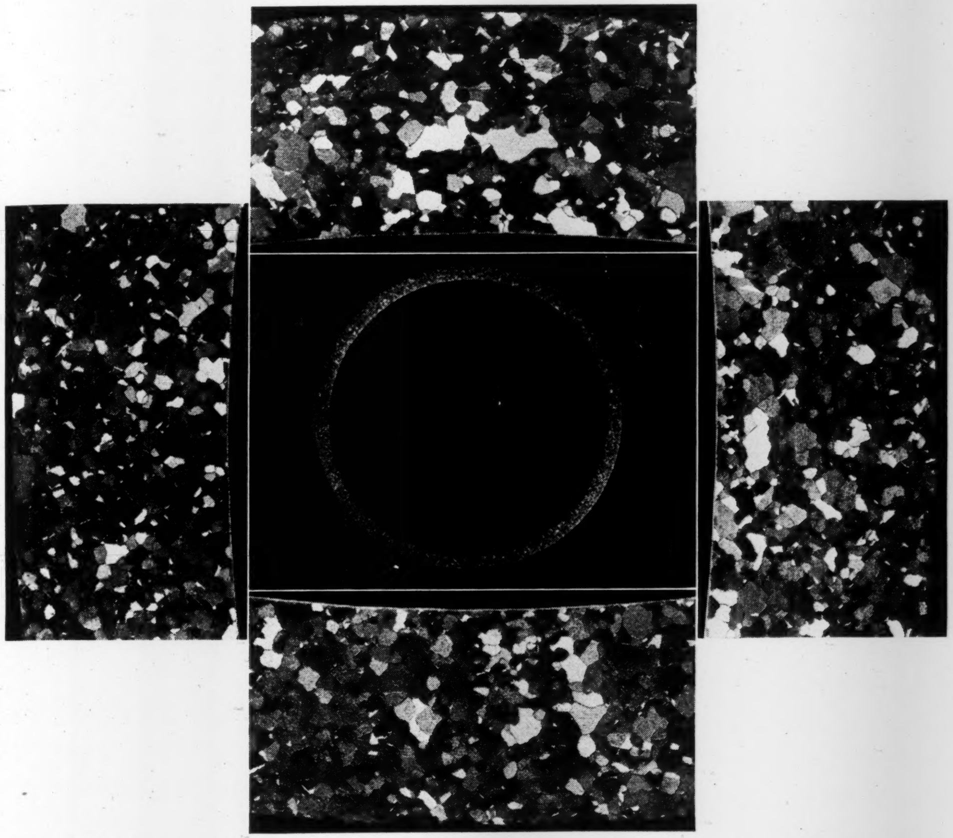

FIG. 16.—Structure of Sample No. 2180, Copper Lead Containing 0.06 per cent Copper and 0.06 per cent Bismuth, Before Testing. Ring (× 1). Micrographs (× 15).

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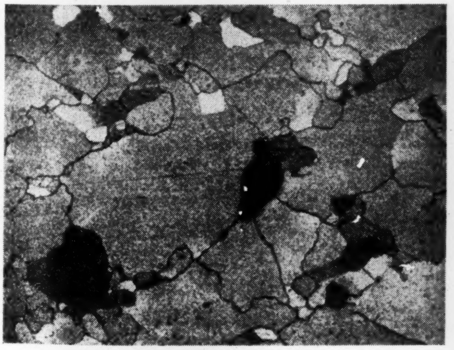

FIG. 17.—Small Intercrystalline Crack in Structure of Sample No. 2180 After Test (× 100).

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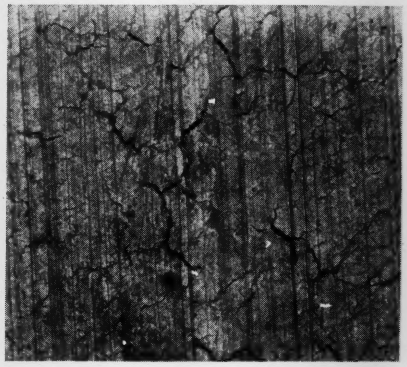

FIG. 18.—Surface Condition of 800 psi. Specimen of Sample No. 2856 After Test, Showing Cracking and Distortion of the Die Scratches.

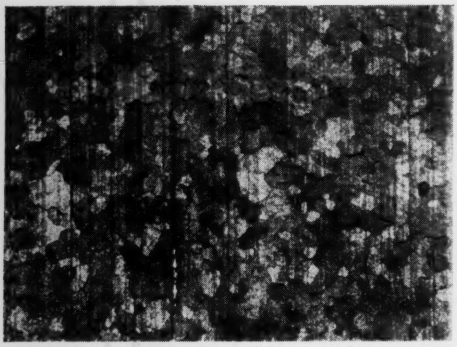

Fig. 19.—Surface Appearance of 800 psi. Specimen of Sample No. 2856 After Test Showing Intercrystalline Nature of Cracks. Surface lightly etched to show structure.

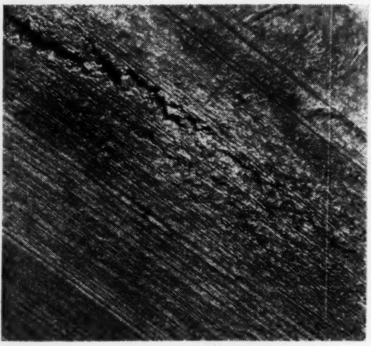

Fig. 20.—Surface Condition Adjacent to Failure of 1500 psi. Specimen of Sample No. 1228 After Test, Showing Intercrystalline Cracking and Distortion of Die Scratches.

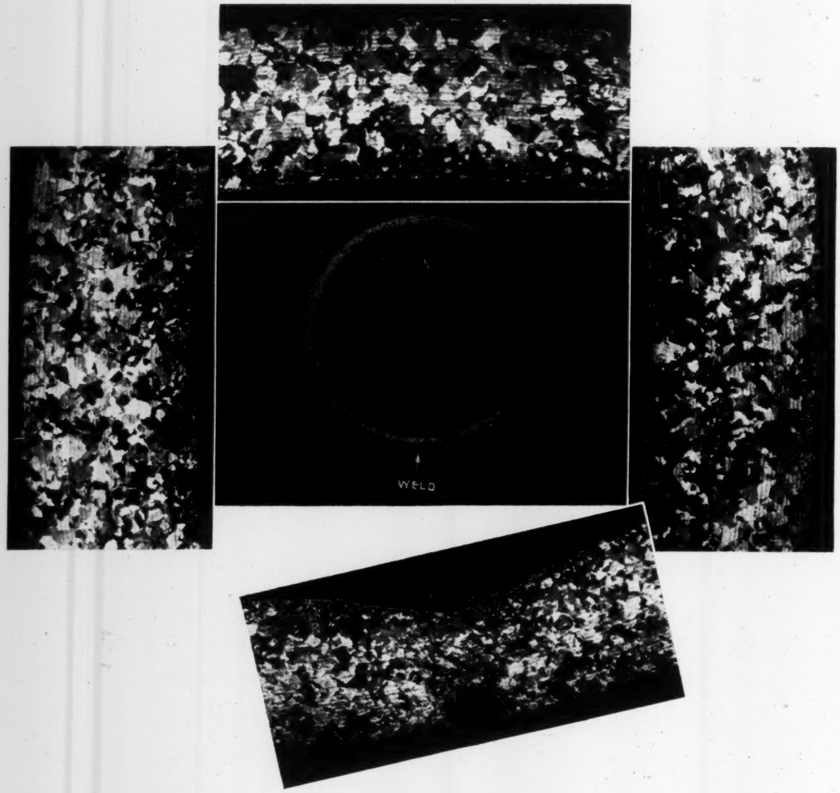

Fig. 21.—Structure of Cross-Section of 1500 psi. Specimen of Sample No. 1228 After Test, Showing Intercrystalline Cracks Around Failure. Ring (× 1). Micrographs (× 15).

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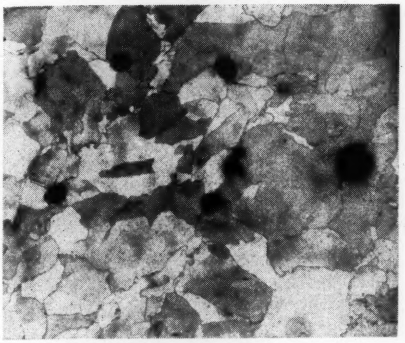

FIG. 22.—Intercrystalline Cracks in Structure of 1500 psi. Specimen of Sample No. 1228 After Test (× 100).

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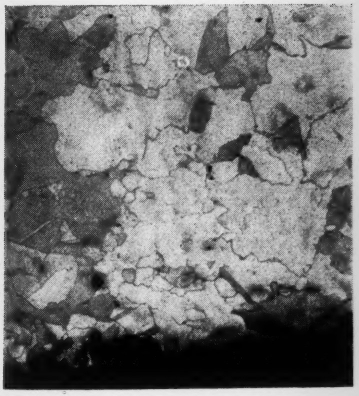

FIG. 23.—Structure of Radial Weld Area at Bottom of Section Shown in Fig. 21 (× 100).

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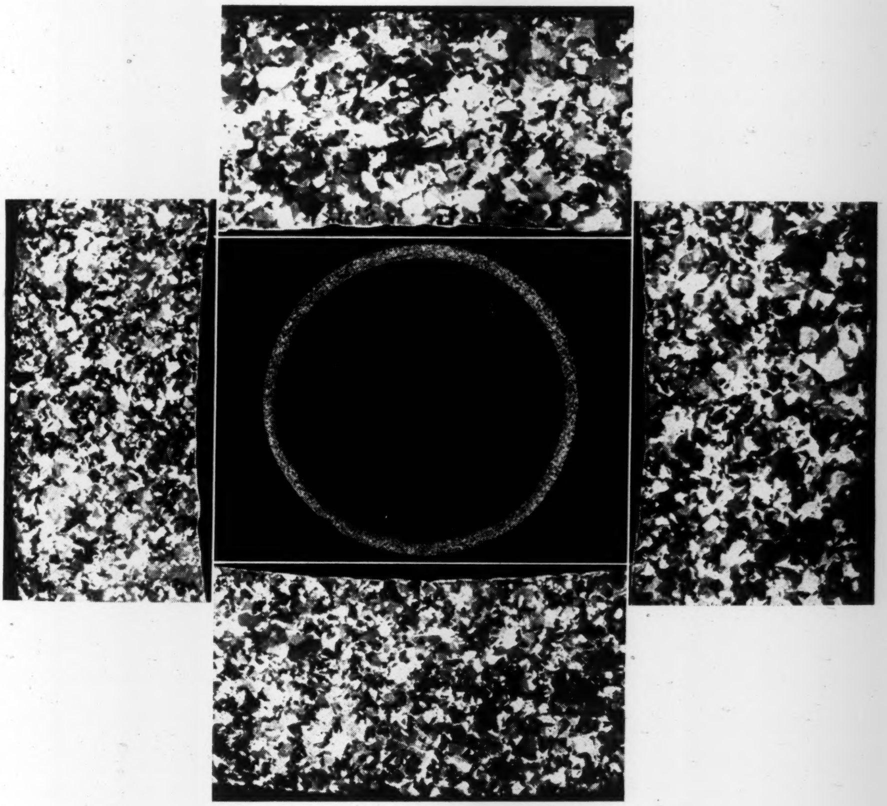

FIG. 24.—Structure of Sample No. 1228, an 0.03 per cent Calcium Lead, Before Testing. Ring (× 1.3). Micrographs (× 20).

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talline cracking than the copper-bearing or common lead samples, especially in areas which had been bent or dented such as sometimes occurred when 250 ft. long tubes, 3 in. in diameter, were tested. This more extensive cracking was attributed to stress corrosion cracking. Many lead alloys are subject to precipitation hardening and it is assumed that electrolytic corrosion occurs as described by Dix (6).

The surface of the 800-psi. specimen of sample No. 2856, after being on test for 174,200 min., was cracked as shown in Figs. 18 and 19. The irregular die scratches in Fig. 18 show the effects of grain rotation in addition to the cracking, whereas the lightly etched surface shown in Fig. 19 clearly illustrates the intercrystalline nature of the cracks. This specimen, which consisted of 270 ft. of pipe, was more or less cracked as shown in Fig. 18 over the entire length.

The surface of the 1500 psi. specimen of sample No. 1228 at the failure also showed irregular die scratches, raised grains and a network of intercrystalline cracks surrounding the failure, as illustrated in Fig. 20. The cross-sectional structure is shown in Fig. 21. Many large cracks in the vicinity of the failure appear in the bottom micrograph. The general grain appearance and size after test was the same as before test when examined at 15 to 20 diameters but examination at 100 diameters showed that the grain boundaries after test had an irregular or fringed appearance. The many small intercrystalline cracks are shown magnified 100 diameters in Fig. 22. Unfortunately the strong Superoxyl etch made the small cracks appear round like holes. Figure 23 shows the structure of the bottom radial weld in this specimen thus eliminating any doubt that these square-edged failures necessarily occur at welds. Figure 24 shows the original structure of the tube.

CONCLUSIONS

The authors have commented briefly on the various methods used for testing lead cable sheathing and have shown the relatively great amount of information obtained from a series of four burst tests on each sample made at a succession of different pressures. Burst tests indicate strength, ductility, creep characteristics, structural weaknesses, and tendency to stress or stress-corrosion cracking.

It was pointed out that the burst life of a lead tube or cable sheath varies as an exponential power of the hoop stress.

A new theory has been advanced that the life of all the common lead alloy cable sheath materials will be approximately the same when they are stressed at a low stress of the order of 400 psi.

It has been shown that when lead materials fail under a long sustained tensile stress that is insufficient to cause appreciable plastic flow, the type of failure will be intercrystalline resembling fatigue. Because the grain boundaries are weaker than the lead grains and because the radial welds offer the shortest possible grain boundary path across the lead wall, it frequently happens that these failures occur at a weld and may be erroneously attributed to inherent defects.

The authors have found the burst test especially useful in development work as it gives in one test all the necessary engineering information except fatigue and corrosion resistance to evaluate lead sheath materials.

Acknowledgment:

The authors wish to express their appreciation to R. L. Duffner and M. F. McGrath, Jr., for their assistance in obtaining the data referred to in this paper.

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JOINT DISCUSSION

INTERNAL HYDRAULIC BURSTING TESTS OF LEAD CABLE SHEATHING
BY PHELPS, GATES, AND KAHN

BURSTING TESTS AND TENSION TESTS FOR LEAD CABLE SHEATHING
BY MOORE, DOLLINS, AND CRAIG

THE TESTING OF LEAD CABLE COVERINGS
BY BASSETT AND SNYDER

MR. HERMAN HALPERIN.[1]—Each of these three papers adds substantially to our knowledge of the properties of lead and lead alloys, a field of knowledge which was practically neglected until about twelve years ago.

With respect to the internal pressures imposed on the sheaths of electric power cables in service, there are three distinct classes. First, in oil-filled cable the internal pressure is nearly constant at relatively low values of generally 10 to 15 psi. Second, in extra-high-voltage cable of the solid type with reservoirs containing thin oil connected to the joints, the internal pressures go through daily cycles. As the cable heats with increasing load, the pressure rises to about 30 psi. normally or on a few rare occasions to as high as 60 or 80 psi. (about 600 or 800 psi. hoop stress). The sheath is expanded slightly and when the load decreases vacuum of 10 or 20 in. of mercury develops. On single-conductor, 66-kv. cable of this kind in Chicago the commercially pure lead sheaths have expanded at an average rate of over 0.2 per cent per year, without decrease in rate in 14 yr. of service. Third, for solid-type cable operated at about 24 kv. and less, the joints are usually filled with a semi-hard compound. The sheath is then pushed out during only the first few loading periods and not much pressure is developed subsequently except during extra-heavy loading.

Most of the troubles in service have been with the sheaths in the second class, but some sheaths in the first and third classes have broken open, too—even some on 4000-v. cable.

Both Mr. Bassett and Mr. Phelps show straight lines on graphs of the log of time to burst plotted against stress with some bends in the lines. As yet, the test results at 500 psi. and less are meager but what data we have seen from Moore's laboratory and other laboratories have suggested that there are further bends in the lines that indicate longer lives at the lower stresses than are predicted in these papers. Furthermore, the vacuum inside the cable during a large part of the service time tends to lengthen the sheath life.

Mr. Bassett concludes that all of the common lead alloys have about the same life at low stress. Data we have studied indicate that calcium-type lead alloys have some advantages both in life to fracture and in rate of creep, even at the lower stresses. On solid-type cables the stronger sheath material will cause greater pressures to develop and so the calcium-lead must be superior at somewhat higher stresses than the commercially pure lead sheath. The calcium-lead appears to be superior also in life to cracking due to bending in manholes.

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[1] Assistant Equipment and Research Engineer, Engineering Dept., Commonwealth Edison Co., Chicago, Ill.

The data on elongation with time in these bursting tests are, of course, similar to the elongation data for strip specimens, both showing three stages of creep. Much of the discussion of these data on elongation is concerned with the total distention to burst. It seems to me that the useful life of the sheath in service *is ended during the second stage* of creep before these curves turn sharply upward as they do in Figs. 9 to 12 of Mr. Phelps' paper, for example. Toward the end of this stage many slightly irregular sheaths would burst and the high rate of trouble would justify replacement of all of the cable at one time. Also, when the sheath expands by *about 2 to 5 per cent*, serious deterioration and perhaps breakdown of the insulation electrically is apt to occur. Data on behavior of the sheaths up to the beginning of the third or final stage of creep seem more significant than data on the elongation to fracture.

It should be noted that most of the sheath openings that have developed in service have been due to nonmetallic impurities or high eccentricity in circumferential thickness. In the past few years the manufacturers have so improved their product that these deficiencies have become almost entirely absent.

Both Mr. Bassett and Mr. Phelps find that the bursting test if conducted at relatively low stresses for long periods of time gives a reliable indication of the quality of the sheath. If the test is hastened by applying high stresses, the indications are not so reliable because nonmetallic inclusions tend to harden the metal and thereby to strengthen it in short-time tension tests. The bending test applied to circumferential strip specimens gives definite information on the quality of the sheath in a very short time, according to our experience with about 250 sheath openings that occurred in cable in service.

In a paper by Greenwood and Werner presented in 1939 before the British[2] Institute of Metals, copper was shown to increase the rate of creep of lead at constant stress if the content was less than 0.05 per cent. The maximum rate of creep was shown to be at about 0.03 per cent of copper. We found that data from Moore's tests were in close agreement with this statement. It would be of interest if others who are making creep tests such as those reported here would check this point too because some people have had the idea that 0.03 to 0.04 per cent copper is just as good as 0.06 per cent.

We were interested in Mr. Bassett's unamplified statement that the results of tests on sheath from cable did not conform to the rule that the character of the fracture changed from a ductile necked-down separation to a brittle, blunt, intercrystalline failure as the stress was reduced. If there is an appreciable difference in characteristics between extruded pipe and sheath removed from cable, it would seem that any "rules" to be applied to cable should be based primarily on tests of the latter.

Mr. Bassett also suggests that some of the sheath openings occurring in service in 66-kv. cables in Chicago may be connected with intercrystalline stress cracking. This does not appear to be the case—at least in the majority of the troubles. In recent years at least half of the openings in these cables have been of the ductile, necked-down type and the percentage of necked-down openings has increased with increasing time in service. Of the remaining openings, part were due to laminations at defective charge welds which could not be connected with intercrystalline stress cracking. In the past 4 yr. only about 25 per cent of the total openings on these cables have been blunt fractures at seam welds and in most of these cases the welds were obviously of poor quality.

---

[2] J. Neill Greenwood and H. K. Worner, "Types of Creep Curve Obtained with Lead and Its Dilute Alloys," *Journal*, Inst. Metals, Vol. LXIV, No. 1, pp. 135–167 (1939).

The agreement between the data obtained in burst tests by Phelps and in tests on strips by Moore is of considerable interest.

MR. LYALL ZICKRICK[3] (*presented in written form*).—The curves that Phelps has shown are very interesting, and show another attempt to predict or gain some clue about expected service life from comparatively short-time tests. Unfortunately, the authors struggling with the difficulty of trying to accomplish two things at once: first, a test for quality and properties of material; and second, an analysis or interpretation of the structural characteristics introduced in cable sheath during manufacture. Confusion arises when these structural characteristics interfere with what would normally be the physical properties of the metal. For example, the test results given in Figs. 11 and 12 and discussed on pages 901–902 cover copper-bearing lead samples of three different compositions evidently from three sources, and at least two different processes of manufacture, with unknown extrusion temperatures, probably hot for the ordinary die block press and cold for the slug press. Yet a conclusion is drawn that the decrease in total stretch with decrease in stress is attributed to failure of welds or flow lines.

The authors make several references to the term "cold die welds." I seriously doubt whether cable manufacturers today encounter any trouble with die welds. Personally, in all the testing I have done in the past 10 yr. I have encountered only one case where the trouble could rightly be attributed to a "cold weld" and that on a small-sized cable of about 1-in. diameter. What are termed flow lines resulting from the area where the two charges join have given varying degrees of trouble, and the structures of these are so different that I feel they should not be so closely associated with die welds. If a sheath has die welds at all, they are present throughout the entire cable length while flow line structures are not.

Regarding the term "flow line structures," such structures are frequently referred to in the paper as laminations. It is unfortunate that there is not in existence a better agreement on definitions. The Insulated Power Cable Engineers Assn. gives the following:

> *Flow Lines.*—Certain lines, mostly circumferential, visible to the naked eye, appear on the prepared surface of a section of extruded lead sheath when etched in a suitable etching fluid. These lines in normal sheaths result from the flow of the lead through the press and die block and are grouped together under the general term "Flow Lines."
>
> *Laminations.*—Laminated lead in the true sense is lead that is formed in layers without proper bond between same. These layers may occur in the form of approximately concentric arcs. Layers so formed can be readily separated and are usually easily determined by visual inspection.
>
> The term "laminated lead" has frequently been misapplied in classifying that which in reality represents flow lines. The presence of such regions has no significance with regard to the strength of the bond between adjacent layers or to the occurrence or nonoccurrence of seams or fissures between the layers.

In the section headed "Failures in Welds" the reader is somewhat misled.

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[3] Metallurgist, General Electric Co., Schenectady, N. Y.

If he reads carefully, he learns that the failures were always in the thinnest part of the wall thickness of the sheath where they would normally occur if no welds were present; also that distinction is not always clear as to whether the failures occurred in the die weld or flow line structure adjacent to the die weld, and whether or not they showed evidence of being intercrystalline.

The difference in type of break experienced between short-time and long-time tests is not new and is frequently encountered. The matter of grain structure alone may have an important bearing on whether ultimate failure occurs at the weld as a matter of weakness or as a matter of coincidence. We find in our long time tests on strip samples tested at room temperature that the change-over to the intercrystalline type of fracture becomes noticeably detectable in the neighborhood of 600-psi. stress. This intercrystalline type of fracture becomes much more pronounced as lower stresses are applied for longer periods of time. The intercrystalline cracks are not always detected at the higher stress, for as the cracking reduces the cross-section, the unit load becomes greater, and the specimen soon reaches a point where failure occurs as a regular necked-down break. A microscopic examination of the area adjacent to the failure will often show the presence of small intercrystalline cracks, especially if the stress has been applied for a period of time.

Because of these and other factors, I wonder whether it is not going to be exceedingly difficult to extrapolate to cable life expectancy from comparatively short-time laboratory test data. The mechanism of intercrystalline cracking in lead is based on such difficult metallurgical theory that it is often overlooked. Further complicate this picture with the facts that: (*a*) lead will recrystallize at room temperature, (*b*) different grain size gives different physical properties, and (*c*) areas which have been slightly cold worked become subject to grain growth; and the problem fast loses its simplicity. Definite conclusions drawn from a study of only one phase of the subject may later prove to have been too hastily formed.

However, it is such work as this that brings sheath problems to the foreground and forms the groundwork that leads to the production of a better lead sheath.

MESSRS. HOWARD S. PHELPS,[4] ALBERT M. GATES,[4] and FRANK KAHN[4] (*Authors' closure by letter*).—The authors are in hearty accord with much of Mr. Zickrick's discussion, particularly those sections which stress the complexity of testing lead or lead cable sheathing. In any problem involving so many known and unknown variables, the researcher must determine if control of individual variables will produce results consistent with that control in spite of other variables that are present. The results referred to by Mr. Zickrick in Figs. 11 and 12 show such an attempt. Results of other tests not reported in this paper substantiate the conclusions drawn from the data presented, that weld or flow line failures reduce the total stretch of lead sheathing samples under internal pressure bursting tests.

Mr. Zickrick's discussion indicates that the authors did not sufficiently stress the fact that "cold die welds" and "laminations" were not involved in the test results reported. On the contrary, one of the primary objects of the paper was to point out that the types of failure previously associated only with "cold die welds" and "laminated lead" do in fact obtain in sheathing represented by the various manufacturers to be as sound as the industry produces.

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[4] Engineer, Special Tester, and Senior Technical Assistant, respectively, Special Investigation and Testing Division, Philadelphia Electric Co., Philadelphia, Pa.

The authors concur with Mr. Zickrick in considering it unfortunate that there is not in existence a better agreement on definitions of "flow lines" and "laminations." The sense of these terms as used in the paper is given in the section entitled "Description of Welds." The definitions given by the Insulated Power Cable Engineers Association are of little help in the understanding that "flow lines" do not result from the flow of homogeneous lead from a single charge through the press and die block, or that inclusions of oxides between charges of lead may appear in the flow lines as "laminations."

The discussion of the section headed "Failures in Welds" calls for some amplification. It is true that the paper does not distinguish between die weld and flow line failures. The reason for this was that in most cases the flow lines were so close to the die weld that it was very difficult to make the distinction. Examination of microstructure by the authors and substantiated by manufacturers disclosed that both die weld and flow line failures do occur. This is the important point from the viewpoint of cable users, but of course the manufacturers must determine what can be done about it.

Similarly, it is not so important to cable users whether die weld or flow line failures are intercrystalline or fail by some other process unknown to the authors. The important point is that these failures reduce the time-to-burst for cable sheathing below that which would obtain if wedge or intercrystalline failures were allowed to develop normally. The authors do not claim that intercrystalline failures are new, but they do not know of any other published report that points out the susceptibility to die weld and flow line failures of cable sheathing generally accepted as being sound.

Although an extensive discussion of the subject of creep is beyond the scope of this paper, it may be well to express a word of caution with respect to Mr. Halperin's interpretation of some of the data given. Unfortunately the method of presenting rate of stretch data shown in Figs. 9 to 12 is not convenient for analyzing changes in rate of stretch. It is the nature of the ratio paper used that any constant rate of stretch will plot as a curve that can be said to "turn sharply upward." When the data of Figs. 9 to 12 are plotted on rectilinear cross-section paper, the curves show surprising differences. Although all the curves for grade III lead and those for copper-bearing lead at the higher stresses definitely indicate the three stages of creep assumed by Mr. Halperin, those for copper-bearing lead at the lower stresses do not conclusively show the initial relatively high rate of stretch expected. In these cases, the curves seem to indicate an increasing rate of stretch throughout the test. Furthermore, when die weld or flow line failures are involved, they sometimes occur before what might be considered the final stage of creep. In view of these observations, it appears to be impracticable to draw general conclusions from data on creep up to the beginning of the third or final stage, as suggested by Mr. Halperin.

MR. J. R. TOWNSEND[5] (*presented in written form*).—It is always desirable to simplify a test to the point where a small controlled specimen will be used under conditions that can be accurately controlled. When this end can be achieved it is possible to test a great number of specimens with the least expenditure of time, space, and money. Under such a method of test it is possible to collect much more data on the significant properties of the material than by any other.

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[5] Materials Standards Engineer, Bell Telephone Laboratories, Inc., New York, N. Y.

Engineers find it necessary, however, to make tests which simulate in kind and in degree the service conditions to which the product in question will be subjected. Such tests are usually very convincing to the user of the finished product. This type of test is always expensive if substantial amounts of data are collected and the tester may be sufficiently deceived by appearances to gloss over many uncontrolled variables. Such a simulated service test is convincing if it correlates with service, but when it does not the situation is disastrous indeed. It is very necessary, therefore, that simple scientific tests which test one controlled variable at a time, be developed. The use of a service type of test is a step in the evolution of a controlled scientific test. The paper by Messrs. Moore, Dollins and Craig, therefore, is a valuable contribution to the testing art since it shows conclusively correlations between the simulated service test and the laboratory controlled specimen and provides a convincing chain of evidence, thus emphasizing the truth of the foregoing.

MESSRS. A. E. WHITE[6] and C. L. CLARK[7] (*presented in written form*).—Engineers concerned with the use of metals at elevated temperatures have often raised the question as to whether or not results obtained from tension-creep or rupture tests could be applied with a suitable degree of accuracy to tubular members. The results of Moore's paper shed light on this question and definitely indicate that such is the case, at least for the particular deformation rates considered and with the metal in a distinctly plastic condition.

The question of deformation rate and the relative degree of plasticity is raised as it is believed that both may be a factor. It should be pointed out, however, that the majority of tube failures occur during overheating, and under such conditions the deformation rates would be comparable to those considered by the authors and likewise the steel would be in a plastic condition.

The fairly good correlation between the degree of elongation up to fracture in both types of test is of interest as it provides an indication at least of the degree to which a tube will bulge before actual rupture occurs. The decreased ductility with increasing fracture time on certain of the lead specimens considered is also typical of certain steels, especially at 900 and 1000 F.

MR. H. F. MOORE[8] (*Author's closure*).—The sole purpose of the paper presented from the University of Illinois was to see whether any correlation was possible between the results of carefully made long-time bursting tests of specimens of sheathing and the results of tension test specimens cut from the same pieces of sheathing. The results obtained so far are few, but for these pieces of sheathing tested the correlation seems good. It is to be hoped that other people will make other correlating tests, or at least will send us pieces cut from the sheath as the specimens subjected to bursting tests. In the latter case it is important to state the temperature of testing.

MESSRS. W. H. BASSETT, JR.,[9] AND C. J. SNYDER[9] (*Authors' closure by letter*).—The importance of data obtained from burst tests run at values below 600 psi. hoop stress was emphasized by Mr. Halperin. We are continuing all of the tests shown in Fig. 7 at lower values of hoop stress and hope to be able to report these results at a later date. The time required for such tests is, however, so great that our present paper can only be a progress report and indicate the trend of the results. It was interesting, however, to note that the values for samples Nos. 2180 and 2264 of 400,000 and 300,000 min., respectively, were obtained after the original lines in Fig. 7 had been drawn. These additional points fell on the preliminary extension of the lines showing that so far there has been no further change in the slope of the lines.

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[6] Director of Department of Engineering Research, and Professor of Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

[7] Research Engineer, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

[8] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[9] Manager, Metallurgical Development, and Engineer, Metallurgical Laboratory, respectively, Anaconda Wire and Cable Co., Hastings-on-Hudson, N. Y.

Mr. Halperin has very kindly reported operating data regarding power cables which show the relation between the laboratory tests and field experience. In this connection it would appear that some 66 kv. cables have expanded approximately 2.8 per cent in diameter in a period of 14 yr. Laboratory burst tests at 600 psi. indicate that cable sheath expands approximately 9 per cent before failure occurs. Our investigations have definitely shown that there is a marked difference in the rate of expansion of specimens removed from cables and samples prepared from plain extruded pipe which has not received the extra working due to stretching over a core. These differences in sheath and pipe specimens are indicated in Figs. 4, 5, and 6. We wish to call especial attention to Fig. 6 where a direct comparison can be made between samples Nos. 2180 and 2747.

It was very pleasing to note in Fig. 8 the agreement of the work conducted by Mr. Phelps and Mr. Moore and the results obtained in our own laboratory. This would seem to indicate that the test data can be duplicated by observers working in different localities under varying conditions.

The low stress portion of the curves is undoubtedly the most interesting. In this region it appears that we are measuring the cohesion between the crystals rather than plastic flow. This would appear to be especially true in the alloys which show a limited amount of creep and burst with very little plastic deformation.

CREEP OF ALUMINUM SUBJECTED TO BENDING AT NORMAL TEMPERATURES

By Joseph Marin[1] and L. E. Zwissler[2]

Synopsis

This investigation deals with an experimental study of an aluminum alloy subjected to pure bending accompanied by creep. Creep deflections were measured for various values of constant bending moments. To interpret these results, tension creep tests were also made for various stress values. With the experimental constants obtained from the tension creep tests, a creep rate - stress relation was established for tension. Using this relation as a basis, a theoretical differential equation was determined for creep deflections in beams. In this way it was possible to make a comparison between the experimental and theoretical values and thus interpret the tests on creep deflections in bending.

Previous Investigations on Creep in Bending

Investigations on creep of metals in bending are few. MacCullough **(1)**[3] tested lead beams subjected to pure bending. By measuring strains at various distances from the neutral axis, experimental proof was obtained that in creep of lead in bending a transverse section originally plane remains plane during bending. A creep relation in tension was established and this relation was assumed to apply for the fibers of the beam specimens in bending. By this means the bending stresses were evaluated from the measured creep strains in the beams. A theoretical bending moment was calculated using these stresses and their distances from the neutral axis. A comparison could then be made with the actual moments applied. For the two specimens tested by MacCullough the calculated values of the moments were 2.8 and 6.3 per cent less than the actual values.

Tapsell and Johnson **(2)** also investigated creep in lead beams subjected to pure bending. The creep strains at different distances from the neutral axis were also measured to show that a plane section before bending remained plane during bending. Tension creep tests were made and a log - log relation was selected between the stress and the creep rate. With this tension creep rate - stress relation the values of the moments were computed and a comparison was made with the actual moments. Two specimens in bending were tested and the differences between the computed and actual moments were 4 and 18 per cent.

Davis **(3)** made tests on steel beam specimens in bending at temperatures of

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 946.

TABLE I.—TYPICAL MECHANICAL PROPERTIES OF ALUMINUM ALLOY 3S-½H.

Tensile strength, psi	21 000
Yield strength (offset = 0.2 per cent), psi	18 000
Elongation in 2 in., per cent	16
Brinell hardness	40
Shearing strength, psi	14 000
Shearing yield strength (offset = 0.2 per cent), psi	11 000
Endurance limit, psi	9 000

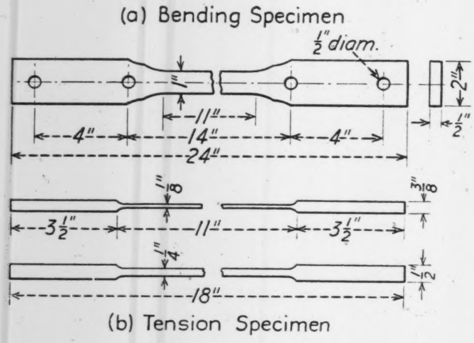

FIG. 1.—Specimens.

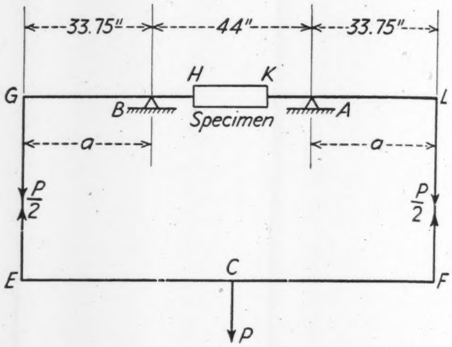

FIG. 3.—Creep Apparatus for Pure Bending.

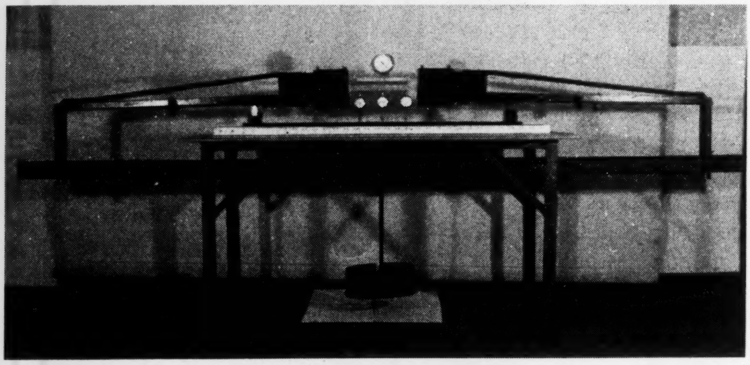

FIG. 2.—Creep Apparatus for Pure Bending.

1500 and 1650 F. Tests on cantilever aluminum beams of constant strength were reported by Sturm, Dumont and Howell (4).

A preliminary investigation on creep of pure lead specimens in pure bending was made by the authors on specimens supplied by the American Smelting and Refining Co. through A. J. Philips. Four specimens 0.5 by $3\frac{1}{2}$ in. in cross-section were tested in pure bending accompanied by creep with moments of 16, 675, 1181, and 1613 in-lb. Creep deflections were measured for an 8-in. length of the beams by Ames dials. During the time of this investigation, however, tension creep test data of lead showed that the creep characteristics were irregular. In view of these results the interpretation of the bending tests could not be made and further investigations on bending in creep of lead were therefore abandoned. This is an important consideration in estimating the significance of the creep tests in bending of lead by MacCullough and Tapsell.

TESTS OF ALUMINUM SUBJECTED TO PURE BENDING

Material and Test Specimen:

The aluminum alloy tested in the present investigation was a common un-heat-treated alloy cold worked to its particular hardness and is designated as 3S-$\frac{1}{2}$H. The mechanical properties of this material, as supplied by R. L. Templin, of the Aluminum Company of America, are given in Table I.

The chemical composition of this alloy is 1.2 per cent manganese with the balance being made up of aluminum and normal impurities.

The specimen for the bending tests is shown in Fig. 1. It was machined from four bars each 2 by $\frac{1}{2}$ in. by 10 ft. The bending specimen was 24 in. in length with a reduced section of 11 in. in the center having a depth of 1 in. and width of $\frac{1}{2}$ in. To determine the variation in the material three static tension control tests were made on specimens machined from each bar. For these tests the yield strength based on 0.2 per cent offset varied from 22,100 psi. to 23,300 psi.

Test Apparatus:

The creep apparatus for bending is shown in Fig. 2. In Fig. 3 this apparatus is represented diagrammatically, showing how a load P produces a pure bending couple equal to $\frac{Pa}{2}$ for all sections of the beam. The specimens were clamped in holders KL and GH (Fig. 2) by means of steel drift pins and steel blocks bearing on the top surface of the specimens by means of two bolts. This permitted the specimen to be securely attached to the holders KL and GH. The holders in turn are supported on a knife edge at A and a cylindrical support at B. The base of the support at B was curved to permit movement in the direction perpendicular to the plane of the specimen. At the ends of the holders KL and GH, load hangers GE and LF were attached. The loading bar EF was supported on these hangers and a load P placed at C in the center of the loading bar produces a pure bending moment in the specimen. Before testing a specimen, the holders and hangers are

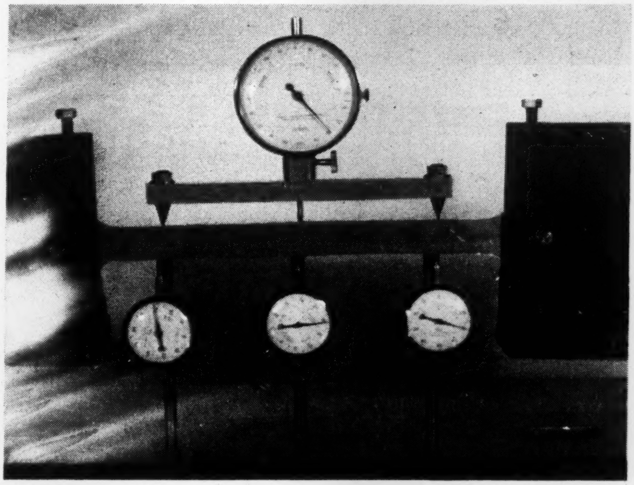

FIG. 4.—Apparatus for Measurement of Creep Deflections.

carefully balanced on their supports by means of adjustable counterweights as shown in Fig. 2. This made it necessary to consider only the weight of the bar *EF* and load at *C* in computing the moment produced. The center creep deflections over a gage length of 8 in. were measured by a Federal dial gage reading to 0.0001 in. This dial was mounted on a rigid bar midway between two knife edges (Fig. 4). Three Ames dials reading to 0.001 in. were also mounted on vertical supports to give readings of deflection on the lower surface of the specimen, one dial being placed at the center and one at each end of the 8-in. gage length. These dial readings furnished a check on the symmetry of loading and a rough check on the deflection values recorded by the top dial. Several test precautions were made such as placing the apparatus on rubber pads, thus preventing effects of vibration. The tests were, furthermore, made in an underground vault in which the temperature variation did not exceed about 3 deg. F.

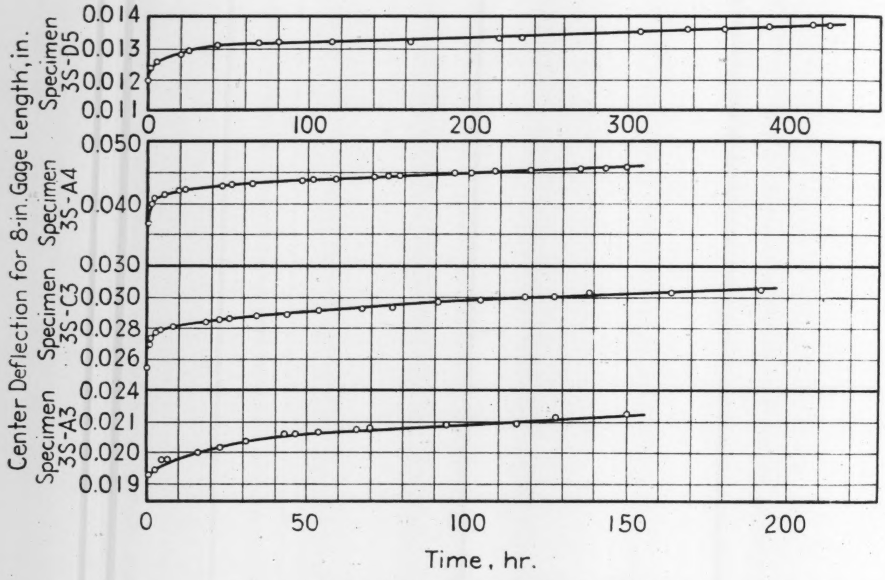

FIG. 5.—Deflection-Time Curves in Bending.

Test Procedure:

Before testing, the cross-sectional dimensions of the specimens were measured at ten sections along the 8-in. gage length. An average value of these dimensions was used for the calculations since the variation was negligible. The loading hangers and holders were then balanced in place by moving the counterweights. The specimen was then mounted in the holders and the initial readings noted on the deflection gage and the three Ames dials. The load to be applied was determined by selecting load values to cover stress values up to the yield point load. The loading bar and load at *C* were then applied, the time of application of load and initial deflection being noted. Readings of creep deflections were then recorded about every $\frac{1}{2}$ min. for the first few minutes. The time interval between deflection readings was gradually increased until after 24 hr. it was only necessary to take readings about twice a day. The three Ames dials were read at intervals of time to check the symmetry of loading. At no time was there a greater difference than ½ division between the end dials.

TABLE II.—CREEP RATE IN BENDING AT 150 HR.

Specimen	Moment, in-lb.	Creep Rate, in. per in. per hr. × 10^6
3S-D5............	687	2.3
3S-A3............	996	5.7
3S-C3............	1333	8.0
3S-A4............	1667	16.7

Test Results:

The test results are shown in Fig. 5 for each bending specimen, giving the relation between the creep deflections and time. The tests covered periods of time up to 400 hr. From Fig. 5 the deflection creep rates for each specimen at 150 hr. were determined and tabulated in Table II.

INTERPRETATION OF TEST RESULTS

In order to interpret the above test results it is desirable to relate the creep rate in bending with that in simple tension. A theory for creep deflections in bending is developed below based on tension creep test results. With this theoretical relation between bending moment and deflection creep rate established, a comparison can thus be made with the above experimental deflection creep rates. It was first necessary therefore, to obtain tension creep data.

TABLE III.—TENSION CREEP TEST DATA.

Specimen	Stress, psi.	Creep Rate, in. per in per hr. × 10^8
3S-B3.2..........	3 472	10.0
3S-D2.3..........	6 227	22.0
3S-D2.2..........	9 655	30.0
3S-D2.1..........	12 321	40.0
3S-B3.1..........	13 130	56.0

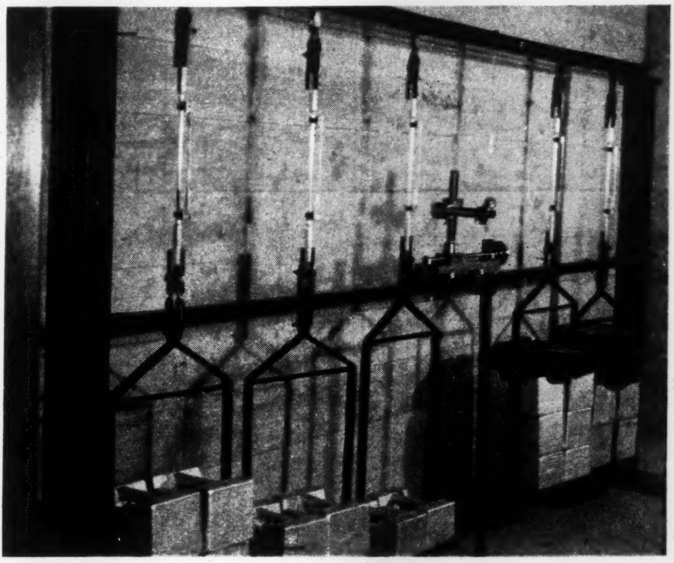

FIG. 6.—Creep Apparatus for Tension.

Tension Creep Tests:

The tension specimen used is shown in Fig. 1. The apparatus for testing is

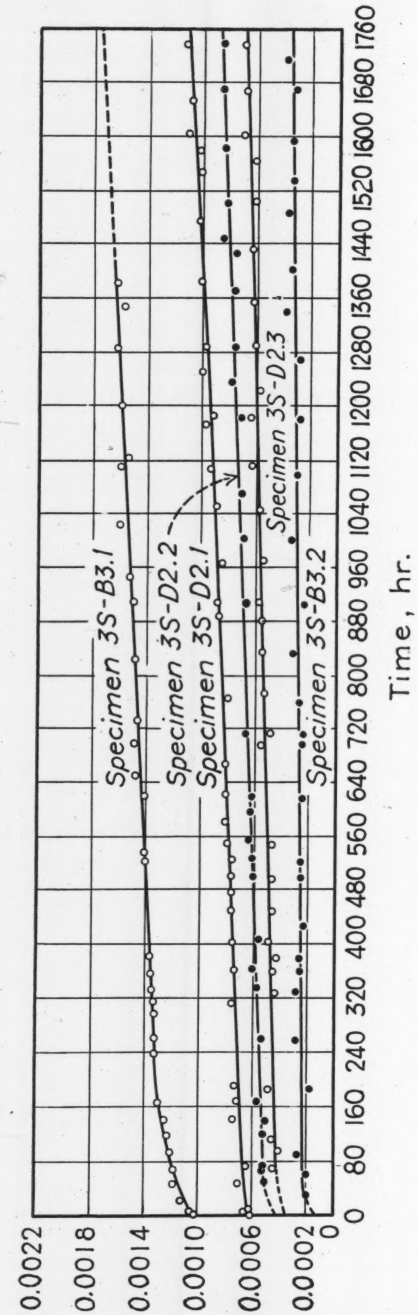

FIG. 7.—Tension Test Creep Curves.

shown in Fig. 6. Five specimens were tested simultaneously at different unit stress values. The specimens were clamped at each end into slotted holders by two Allen set screws. The top holder is attached to the supporting frame by means of an attachment having two bolts at right angles to each other, thus eliminating eccentricity of loading. The lower clamp is fixed in a similar manner to free hanging weights. A gage length of 9 in. was used for measuring the creep deformations. These deformations were measured as shown in Fig. 6 by measuring the movement between the lower end of a rod, attached to the top gage point, and a brass scale attached to the lower gage point. The amount of creep elongation was observed by the micrometer microscope shown.

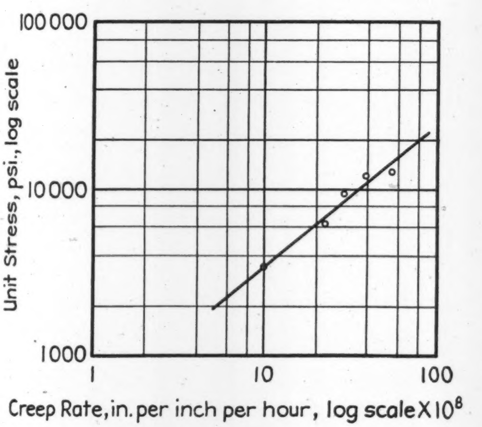

FIG. 8.—Log-Log Relation Between Creep Rate and Stress.

In testing a specimen it was first placed in the upper holder with the strain gage in place. An initial creep strain reading was then recorded with the micrometer microscope. A predetermined load was then applied by means of the load hanger and creep readings observed at intervals of $\frac{1}{2}$ min. The time intervals were gradually increased until readings were recorded only daily.

In Fig. 7 data are plotted for five tension specimens showing the relation between the unit creep strain and time. The unit stress values and corresponding creep rates at 150 hr. are listed in Table III.

Creep-Rate Stress Relation for Tension:

Various procedures and methods of interpretation have been proposed for expressing stress-creep relations in tension (5). The application of these methods of interpretation to the above tension data showed that the log-log method was the most satisfactory. This consists in assuming a linear relation between the log of the creep rate and the log of the stress. In Fig. 8 a log-log relation is shown between the creep rate and the stress. It is apparent from this plot that for these data a linear relation is a good approximation. The equation defining this relation between the creep rate C and stress σ is:

$$C = A\sigma^n \ldots\ldots\ldots\ldots (1)$$

where A and n are experimental constants. For the material tested $A = 6.51 \times 10^{-12}$ and $n = 1.18$.

With a creep-stress relation in simple tension established it is now possible to develop a theory for the deflections in the case of bending accompanied by creep.

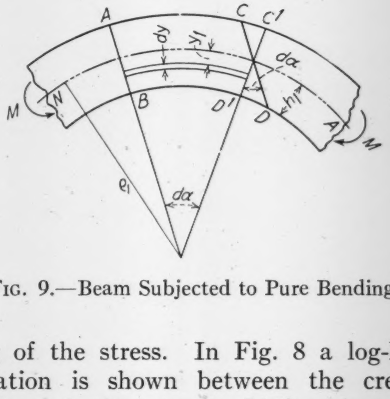

FIG. 9.—Beam Subjected to Pure Bending.

Theory for Creep Deflections in Bending:

To formulate a theory for deflections in the case of bending with creep, consider the section of a beam subjected to pure bending moments M as shown in Fig. 9. The cross-section of the beam will be considered to have one axis of symmetry which lies in the plane of loading. It will be assumed that a plane section such as CD remains plane during creep and deforms relative to the plane AB such that its new position is $C'D'$. Experimental support was obtained for this assumption bynoting that a variation in the position of the deflection gage along the length of the deflected beam produced differences in deflection which could not be measured with a 0.0001-in. dial. This affords an over-all check on the assumption of plane section. The unit creep deformations are then proportional to the distances from the neutral axis and at distances h_1 and y_1 their values are, respectively:

$$e = \frac{h_1}{\rho_1} \quad \text{and} \quad e_1 = \frac{y_1}{\rho_1} \ldots\ldots (a)$$

where ρ_1 is the radius of curvature due to creep. An examination of the creep-time curves for tension in Fig. 7 shows that after about 100 hr. the creep-rate becomes approximately constant. Assuming a constant creep rate, the creep rates for the fibers at distances y_1 and h_1 are:

$$C = \frac{e}{t} \quad \text{and} \quad C_1 = \frac{e_1}{t} \ldots\ldots (b)$$

where t is time in hours. If the creep rate - stress relation developed above for tension applies to the individual fibers in bending, then:

$$C = A\sigma^n \quad \text{and} \quad C_1 = A\sigma_1^n \;\ldots (c)$$

Solving for the creep strain from Eqs. (b) and (c):

$$e_1 = A\sigma_1^n t \ldots\ldots\ldots\ldots (d)$$

Equating the values for the creep strain from Eqs. (a) and (d):

$$\sigma_1 = \left(\frac{y_1}{At\rho_1}\right)^{\frac{1}{n}} \ldots\ldots\ldots (e)$$

This gives a relation for the bending stress at a distance y_1 from the neutral axis in terms of the curvature. For equilibrium, however, the external and resisting moments are equal or:

$$M = \int y_1\sigma_1\,da$$

$$= \left(\frac{1}{At\rho_1}\right)^{\frac{1}{n}}\cdot \int (y_1)^{\frac{1}{n}+1}\,da \ldots (f)$$

In order to integrate Eq. (f) the position of the neutral axis must be defined. This is stated by noting that the summation of stresses over a cross-section of the beam must be zero or:

$$\int_{h_1}^{h_2} \sigma_1\,da = 0 \ldots\ldots\ldots (g)$$

Equations (f) and (g) completely define the curvature ρ_1 due to creep for a moment M at a time t. For a *rectangular cross-section* with width b and depth h, Eq. (g) shows that the neutral axis and centroidal axis coincide, if it is assumed that the creep in tension and compression are equal. Then from Eq. (f):

$$\frac{D}{t}\cdot\frac{1}{\rho_1} = M^n \ldots\ldots\ldots\ldots (2)$$

$$\text{where} \quad D = \frac{1}{A}\cdot\frac{(2b)^n\left(\frac{h}{2}\right)^{2n+1}}{\left(2+\frac{1}{n}\right)^n} \ldots\ldots (h)$$

It is to be noted that the value of D in Eq. (h) is for a rectangular section. Equation 2 expresses the creep curvature in terms of the moment. The relation between the curvature and deflection is for small deflections:

$$\frac{1}{\rho_1} = \frac{d^2y}{dx^2} \ldots\ldots\ldots\ldots (i)$$

where y is the creep deflection at a distance x from the origin. Substituting the value of ρ_1 from Eq. (i) in Eq. 2 gives the differential equation for the creep deflection in terms of the moment: namely,

$$\frac{D}{t}\cdot\frac{d^2y}{dx^2} = M^n \ldots\ldots\ldots (3)$$

Equation 3 is similar to the elastic case of bending. When $n = 1$ and $\frac{D}{t} = EI$, Eq. 3 reduces to the elastic case of bending. The quantity D is independent of M, so for a time t Eq. 3 is a differential equation with constant coefficients. The solution of this relation for various type beams is a matter of direct integration as in the elastic case. Problems, however, involving values of moments with more than one term are best solved by methods of superposition.

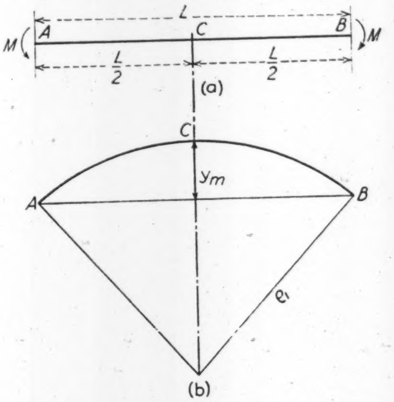

FIG. 10.—Deflection of a Beam Subjected to Bending.

For the case of pure bending used in the experiments, the deflection y_m, (Fig. 10), in terms of the curvature is:

$$y_m = \frac{l^2}{8\rho_1} \ldots . (i)$$

In terms of the moment the deflection is from Eqs. (i) and (j):

$$y_m = \frac{t}{D}\left(\frac{l^2}{8}\right)M^n \ldots\ldots (4)$$

The value of the theoretical center deflection y_m for a gage length L and moment M is completely defined by Eq. 4. Dividing both sides of Eq. 4 by t

$$C_m = \frac{y_m}{t} = \frac{l^2}{8D}\cdot M^n \ldots\ldots (5)$$

The theoretical creep rate C_m is defined by Eq. 5 in terms of D, a function of the material and dimensions, the gage length L, the moment M and the experimental constant n. It is now possible to compare the theoretical and experimental values of these creep rates.

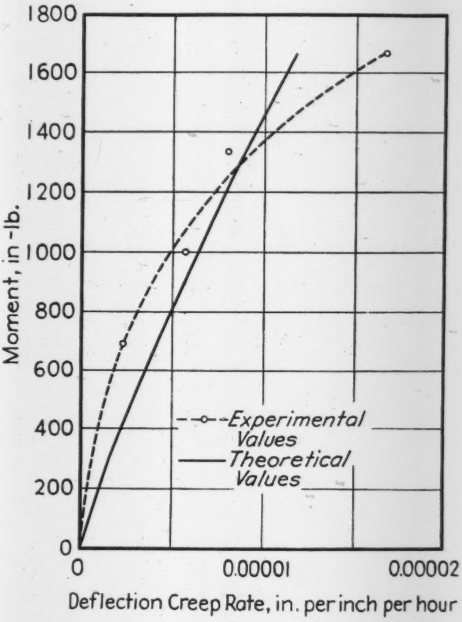

FIG. 11.—Comparison of Theoretical and Experimental Deflection Creep Rates.

Comparison of Experimental and Theoretical Deflection Creep-Rates in Bending:

For the beams tested there was not sufficient variation in the cross-sectional dimensions to justify using separate values. Average values for b and h

were $b = 0.5002$ in., $h = 1.0012$ in. Substituting these values and $L = 8$ in., $A = 6.51 \times 10^{-12}$, $n = 1.18$ in Eq. 5 the theoretical relation between the deflection creep rate and moment is:

$$C_{\dot{m}} = 1.84 \times 10^{-9} M^{1.18} \quad \text{. . . . (6)}$$

Equation 6 is shown in Fig. 11 by the solid line. To compare the theoretical creep rates with the experimental values there is included in Fig. 11 the experimental values as given in Table II.

TABLE IV.—COMPARISON OF THEORETICAL AND EXPERIMENTAL DEFLECTION CREEP RATES.

Specimen	Moment, in-lb.	Creep Rates at 150 hr., in. per inch per hr. × 10⁶		Percentage Difference
		Experimental	Theoretical	
3S-D5.......	687	2.3	4.0	42
3S-C3.......	996	5.7	6.3	9
3S-A4.......	1333	8.0	9.0	11
3S-A3.......	1667	16.7	12.0	39

It should be noted that the comparison is made of creep rates for an elapsed time of 150 hr. The percentage difference between the theoretical and experimental values of the creep rates as determined from Fig. 11 are listed in Table IV.

The large percentage differences between the creep rates for specimen 3S-D5 is to be expected in view of the small values of measured creep obtained due to the low bending stress which was produced in this test. For specimen 3S-A3, the discrepancy between the test values and theory may be due to the fact that the stress produced in the specimen was close to the yield point of the material. For the remaining specimens there is a fair agreement between test results and theory.

In evaluating these results the preliminary nature of this investigation must be considered. The dotted curve in Fig. 11 represents a semiempirical theory based on determining the constants D and n in Eq. 5 by passing the curve through the test points representing the lowest and highest bending moment values.

CONCLUSION

Creep deflections in bending for the aluminum alloy tested were measured on four specimens at various stress values. A reasonably good agreement was found between these values and those obtained theoretically based on a theory developed using tension creep test data as a basis.

Acknowledgments:

The authors wish to express their thanks to R. L. Templin, Chief of Tests of the Aluminum Company of America, for helpful suggestions and for the aluminum used in these tests. The experimental part of this investigation was taken from a Masters Thesis by L. E. Zwissler, performed under the direction of Joseph Marin.

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(5) J. Marin, "A Comparison of the Methods Used for Interpreting Creep Test Data," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I, p. 258 (1937).

DISCUSSION

Mr. J. M. Lessells.[1]—The interesting thing about the paper to my mind is that it brings out another method of obtaining creep data. Most of our knowledge on creep has come from tests in tension, and if we had to begin all over again, we probably would not start with tension for creep studies but might go to the torsion test or the bend test. It is to be hoped that the authors will continue this work because it may lead to increased knowledge of the subject. It may be advisable for the authors to examine the equations used to see whether these apply, since there may be some error in applying the usual equations for bending for high-temperature conditions.

Mr. Joseph Marin.[2]—Regarding Mr. Lessells' comment on the application of "the usual equations for bending," there is apparently a misunderstanding on this matter since an entirely new theory was developed in this paper. This theory is not based on Hooke's law as is the case in the usual theory.

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ANODIC COATINGS SEEN THROUGH THE MICROSCOPE

By F. Keller[1]

Synopsis

The microscope has been found very useful for studying the features responsible for differences in the properties and appearance of the oxide coatings on anodically treated aluminum alloys. This paper describes the various metallographic methods which have been developed for the investigation of anodic oxide coatings and illustrates the results obtained by the use of these methods.

The eye can readily appraise the decorative characteristics of an oxide coating on aluminum but only the microscope can show the detail which determines its appearance. Although aluminum oxide is a transparent, colorless material, anodic oxide coatings on commercial aluminum or its alloys may show color and even a pattern-like structure which are interesting in a scientific way, and which are of real commerical importance. The use of both old and new techniques with the microscope has been invaluable in relating composition and structure to the appearance and other properties of these coatings.

In this country, the most important and widely used method for producing these coatings is known as the Alumilite process.[2] The commercial importance which these coatings have acquired has stimulated interest in all phases of their production, testing, and utilization. In brief, when aluminum is made the anode in a suitable electrolyte, an oxide coating is formed on the surface. This oxide film is essentially Al_2O_3 (**1**).[3] It may also contain small amounts of substances absorbed from the electrolyte as well as oxidation products of the microconstituents or the constituents themselves. The usual methods for examining metals, however, have not been altogether adequate for the investigation of oxide coatings; therefore, new methods have been developed for this purpose. These methods have been described by Keller and Wilcox (**2**).

Methods of Examination:

The metallographic methods now available for the examination of oxide coatings are:

1. Examination of polished *cross-sections* by vertical illumination, dark field illumination, and by polarized light.
2. Examination of *surface sections* of the uncoated metal which are given a metallographic polish and then coated by an anodic oxidation treatment.
3. Examination of *thin sections* of the *oxide coating* itself by means of either ordinary or polarized light.

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[1] Metallurgical Division, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] Protected by patents owned by the Aluminum Company of America.

[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 958.

Ordinarily, polished cross-sections of oxide coated samples are examined under the microscope to determine the thickness, uniformity, and continuity of the oxide coating and the features of the alloy which might influence the oxidation characteristics.

Methods of Preparing Specimens:

The method for preparing samples for this type of examination has been described in detail (3); a summary of the method is as follows:

FIG. 1.—Cross-Section of 2S Aluminum Sheet Which Had Been Oxidized in a Sulfuric Acid Electrolyte for 30 min. The dark band at the top is the oxide coating on one surface of the sheet (× 500).

Reflected light, vertical illumination, unetched.

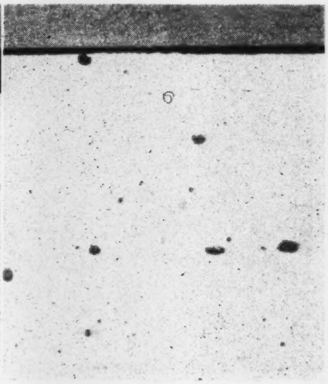


FIG. 2.—Oxide Coating on a Sample of 17S-T Sheet Which Had Been Oxidized in a Chromic Acid Electrolyte for 30 min. (× 500).

Reflected light, vertical illumination, unetched.

A composite specimen is made by taking two or more pieces of the coated sheet, each about 1 by $\frac{3}{4}$ in., and forming a pack with spacers of thin aluminum sheet between each coated sample. The pack is usually drilled and is clamped together with two machine screws. Crevices between the pieces should be avoided but care should be exercised in clamping them together as too much pressure will fracture the oxide coating. The spacers are used to prevent rounding off the edges of the coating during polishing. Molded mountings of Lucite, bakelite or other plastic materials are satisfactory, provided that the oxide-coated surface is backed up with an aluminum strip. In such mountings, the edges of the oxide coating will round off or polish away unless an aluminum back-up strip is used.

The composite or mounted sample of oxide-coated aluminum may be polished by the same technique employed for bare aluminum (4). This method consists of the following operations:

(*a*) First a plane surface is obtained by rubbing the specimen on a medium mill file and on Nos. 0, 00, and 000 metallographic papers in the order stated. These papers should be coated with kerosine or a mixture of kerosine and paraffin.

(*b*) Then, the specimen should be polished on a rotating disk operating at about 300 r.p.m. using a Kitten's Ear broadcloth pad and a suspension of No. 600 alundum flour in water.

(*c*) Finally, the specimen should be polished on a rotating disk covered with Kitten's Ear broadcloth operating at about 150 r.p.m. Heavy magnesium oxide powder is used on the pad which is kept moist with distilled water.

When the polishing is finished, the specimen should be washed in warm water and blown dry. The polished surface should not be touched or rubbed against anything; otherwise the polish will be spoiled. Etching treatments are seldom used on oxide-coated specimens because they tend to pit and dissolve the coating. Owing to differences between the optical properties of the oxide and aluminum, it is easy to differentiate between the coating and the metal without any etching treatment. Where it is necessary to examine the microstructure of the coated alloy in addition to the coating, the sample should be examined in the unetched condition for the characteristics of the oxide coating and again after subsequent etching to observe the microstructure of the aluminum alloy.

FIG. 3.—Oxide Coating on 2S Sheet as It Appears when Examined Under Reflected Polarized Light with Crossed Polarizers. Compare with Fig. 1 (× 500). Unetched.

For the examination, the standard metallurgical microscope may be employed. The coating thickness can be determined very accurately by using a calibrated filar micrometer or an ordinary micrometer eyepiece or by measurements of the magnified image of the coating on the ground glass or a screen.

Examples of how an ordinary cross-section of oxide-coated sheet looks under the microscope are shown in Figs. 1 and 2. The first micrograph shows the appearance in cross-section of the oxide coating on commercial 2S sheet which had received a certain Alumilite treatment in sulfuric acid electrolyte for 30 min. The oxide coating on this sample is about 0.0004 in. in thickness. The second illustration shows the oxide coating on a sample of 17S-T alloy sheet which was treated in accordance with Navy Department Process Specification SR-19c. This treatment involves oxidation for 30 min. in an electrolyte containing from 5 to 10 per cent of chromic acid.

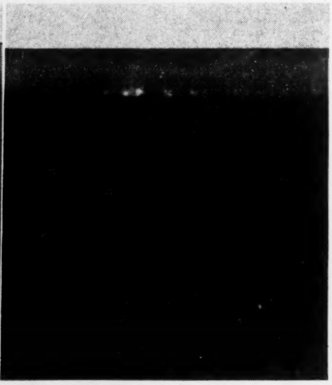


FIG. 4.—Change in Appearance of the Coating Illustrated in Fig. 3 by a Sealing Treatment in a Solution Containing Potassium Dichromate (× 500).

Reflected polarized light with crossed polarizers. Unetched.

Previous work with polarized light has shown that oxide coatings formed in different electrolytes have different optical properties (5). Thus, the use of polarized light may aid in the identification of the oxide coating. The oxide coating on the aluminum sheet illustrated in Fig. 1 appears somewhat different when examined by polarized light with crossed polarizers. This is shown by Fig. 3. The change in appearance which results when this coating is sealed in a solution containing potassium dichromate is shown by Fig. 4. Under the microscope, the sealed coating appears bright yellow with polarized light, whereas the coating on the unsealed sample has a light brown color.

Polished and Oxidized Metallographic Samples:

The effect of variations in microstructure upon the appearance of the oxide coating can be determined by the examination of the surface of polished and anodically oxidized samples. Specimens for this type of examination are polished in the same manner as for ordinary examination. Before examination, however, they should be given the desired oxidation treatment. The anodic coating formed on such a sample is ordinarily very smooth; consequently, the characteristics of the coating and of the alloy at the interface can be observed by examination of the surface of the sample. Either vertical or dark field illumination can be used. The structure obtained with this method of preparation is illustrated in Fig. 5.

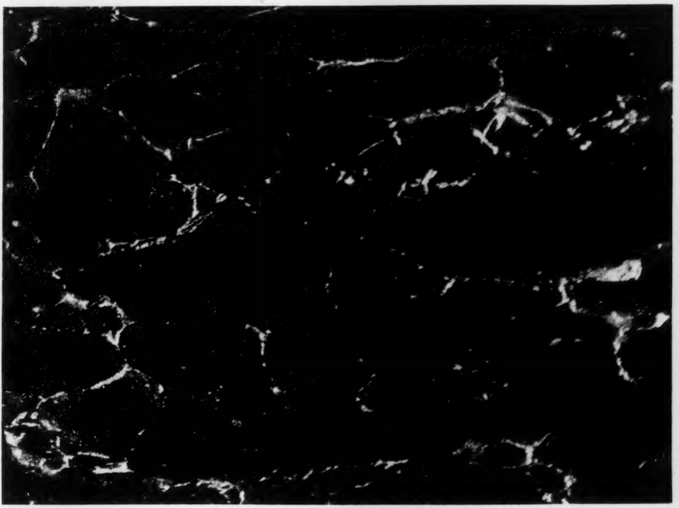
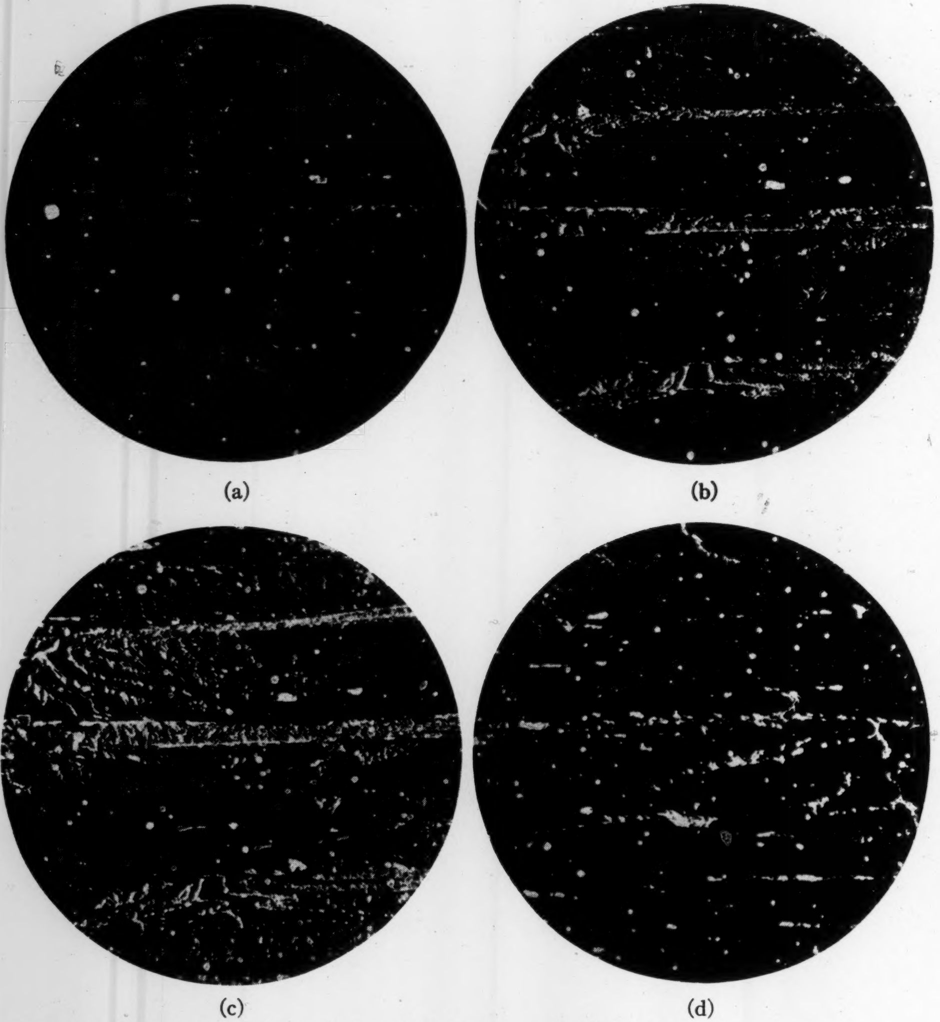


FIG. 5.—An Aluminum-Magnesium-Zinc Alloy Which Was Given a Metallographic Polish and Then Was Anodically Coated in a Sulfuric Acid Electrolyte. The structure shown is that of the oxidized surface as it appeared with dark field illumination (× 15).

Once the anodic oxidation of a sample has been started, oxide continues to form between the metal and the previously formed oxide; thus, the oxide coating already formed is being pushed away from the metal-oxide interface continuously during the oxidation process. Under these conditions, oxidation products from the constituents and some of the constituents themselves may be occluded in the oxide coating. It has been shown by previous work (2) that the amount and distribution of these oxidation products and constituents in the oxide coating have an important influence upon the color, appearance, hardness, and porosity of the oxide coating. A non-uniform distribution of the constituents and their oxidation products in the oxide coating causes the streaking sometimes encountered on anodically coated aluminum alloys. The amount and distribution of the products which remain in the oxide coating are related to the microstructure of the material, the duration of the oxidation treatment, and the conditions of oxidation.



(*a*) Before oxidation. Dark field illumination, etched in ½ per cent hydrofluoric acid.
(*b*) Oxidized for 10 min. in sulfuric acid electrolyte. Dark field illumination, unetched.
(*c*) Sample oxidized for an additional 20 min. Dark field illumination, unetched.
(*d*) Oxide coating removed by a solution which did not attack the aluminum. Dark field illumination, unetched.

FIG. 6.—Constituent Arrangement on the Polished Surface of a 99.75 per cent Aluminum Sheet (× 100).

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It is to be expected that there would be more particles of constituent and more oxidation products occluded in a thick oxide coating than in a thin coating. This accumulation of constituents and oxidation products in the oxide coating is illustrated by Fig. 6. The constituent arrangement on the polished surface of a sample of 99.75 per cent aluminum sheet before oxidation is shown by Fig. 6 (*a*). After this sample was oxidized for 10 min. in a sulfuric acid electrolyte, the structure of the same surface area appears to contain more constituents as shown by Fig. 6 (*b*). A further accumulation of products in the oxide coating occurs when the same specimen is oxidized for an additional 20 min. (Fig. 6 (*c*)). More evidence that the features which influence the appearance of the oxide coating are in the coating and not at the interface is obtained when the oxide coating is removed by immersion in a solution which does not attack the aluminum. Such a treatment removes the products responsible for the difference in appearance and transparency between the coatings shown in Fig. 6 (*b*) and (*c*). The appearance of the stripped sample is shown in Fig. 6 (*d*).

It is apparent from these illustrations that examinations of anodically coated metallographic specimens reveal the microstructural features responsible for streaks and variations in the surface appearance of oxidized aluminum samples. These features cannot be observed by removing the oxide coating from a streaked or discolored area and examining the microstructure of the metal.

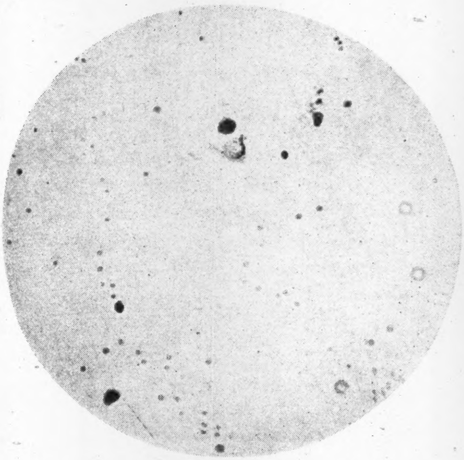

FIG. 7.—A Thin Section of the Oxide Coating on a Sample of 99.95 per cent Aluminum Sheet as It Appeared when Examined by Light Transmitted Through the Coating. Oxide is very transparent (× 100).

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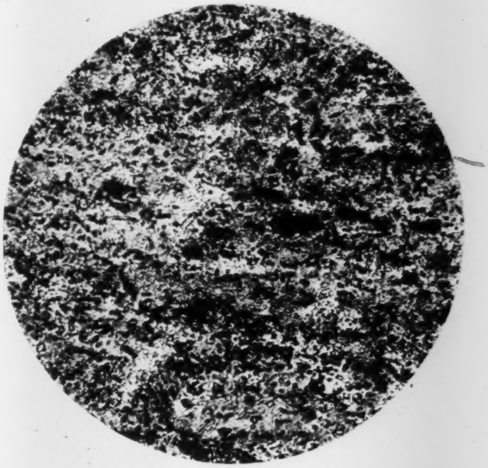

FIG. 8.—A Thin Section of the Oxide Coating on a Sample of 99.2 per cent Aluminum Sheet (× 100).

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The oxide coating contains oxidation products of constituents and particles of constituents. It is not nearly as transparent as the coating on high purity aluminum. Transmitted light.

Examination of Thin Sections:

Another method of examining oxide coatings comprises the preparation of thin sections of the oxide coating itself. Specimens of this type are prepared by mounting the sample in Lucite in such a manner that the aluminum can be removed from the back of the oxide coating by grinding on metallographic emery papers until a suitable area of the coating is exposed. Then the exposed area is polished with No. 600 alundum and magnesium oxide until a smooth surface is obtained. Finally, the opposite face of the Lucite mounting is polished on a plane parallel to that of the oxide. Such thin sections can be examined by transmitted light the same as petrographic sections of rocks and minerals. Examination of these thin sections has revealed that the coating on an oxidized aluminum alloy has a characteristic structure. This structure is related to the microstructure of the metal from which the oxide coating is formed. By this method of examination, much useful information has been obtained regarding the color, streaking and transparency of oxide coatings.

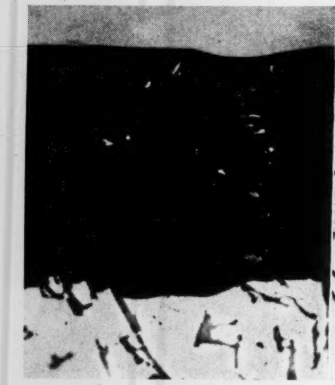


FIG. 9.—Oxidation Characteristics of Silicon Constituent (× 500). Vertical illumination, unetched.

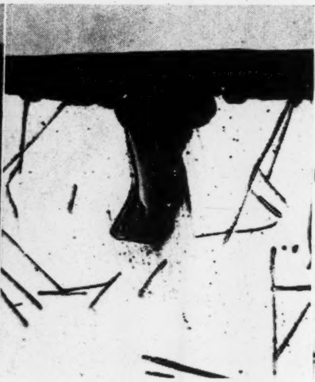


FIG. 10.—Oxidation Characteristics of $FeAl_3$ Constituent (× 500). Vertical illumination, unetched.

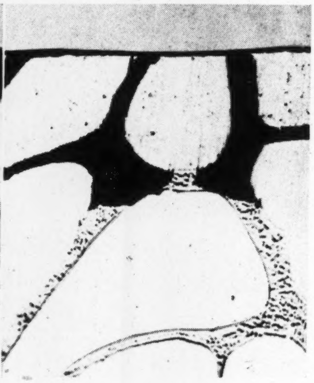


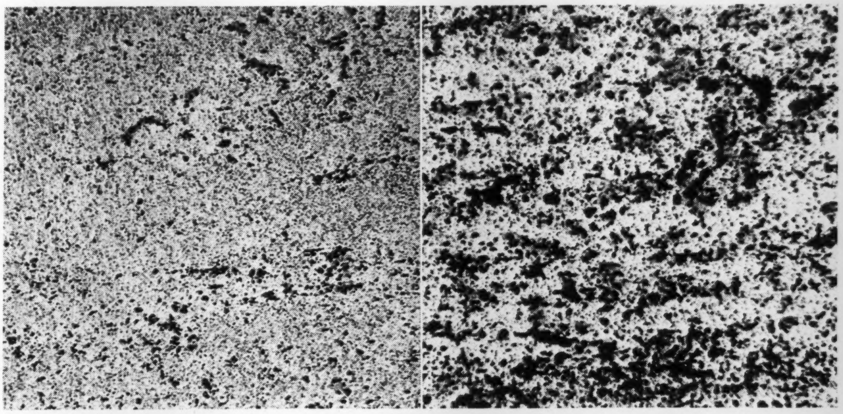
FIG. 11.—Oxidation Characteristics of $CuAl_2$ Constituent (× 500). Vertical illumination, unetched.

The oxide coating on high purity aluminum (99.95 per cent) is very transparent and shows practically no structure as illustrated by the thin section shown in Fig. 7. For comparison, the structure of a thin section of an alloy which contains several different constituents (99.2 per cent aluminum) is shown in Fig. 8. In this illustration, the structure of the oxide coating is shown clearly. This structure is derived from the microstructure of the metal from which the oxide was formed.

Effect of Alloying Elements:

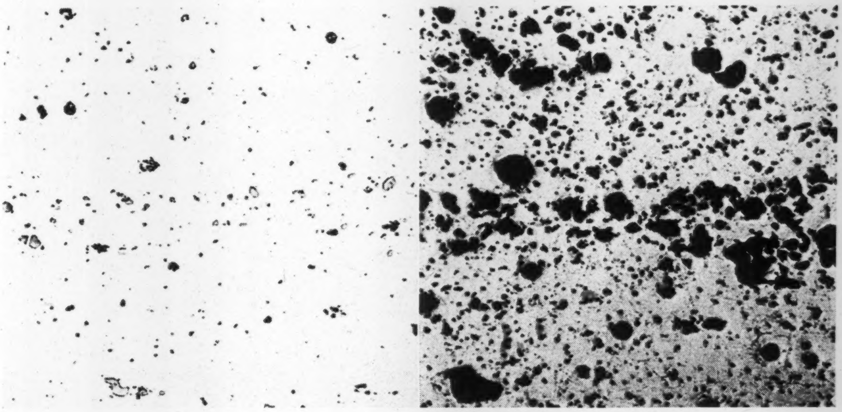
The Alumilite process is used for the commercial treatment of aluminum alloys as well as pure aluminum. When other elements are present in aluminum as impurities or are added to obtain certain characteristics, they may be in several different forms. These elements may be in solid solution, they may form intermetallic compounds which can be put into solid solution by suitable thermal treatments, or they may form intermetallic compounds which are insoluble in solid aluminum. These compounds as microconstituents form the structure which is revealed under the microscope. The character of this structure is influenced by the composition of the alloy, the solidification conditions, the working conditions, and by the thermal treatments the alloy receives.

Fundamental investigations have shown that the different constituents have different oxidation characteristics (6). A few are not dissolved or oxidized during the Alumilite treatment; consequently, they remain in the oxide coating and affect the color and appearance. An example is the silicon constituent shown in Fig. 9. Some other constituents are dissolved or oxidized either at the same rate or more rapidly than the aluminum matrix; an example ($FeAl_3$) is shown in Fig. 10. Colored products from some of these constituents may remain in the coatings and cause streaking and discoloration. A third type of constituent is either oxidized or dissolved completely during the oxidation process and at a more rapid rate than the aluminum matrix. On alloys containing these constituents out of solution in substantial amounts, thin and rough coatings result. The oxidation characteristics of this type of constituent are illustrated by $CuAl_2$ as shown in Fig. 11.



(*a*) In the annealed temper. (*b*) Oxidized in a sulfuric acid electrolyte.

FIG. 12.—A Surface Section of an Aluminum-Magnesium-Silicide Alloy (53 S-O). Vertical illumination (× 100).



(*a*) In the heat-treated temper. (*b*) Oxidized in a sulfuric acid electrolyte.

FIG. 13.—The Surface of a Sample of an Aluminum-Magnesium-Silicide Alloy (53S-T). Vertical illumination (× 100).

Some alloying additions cause a coloring of the oxide coating when they are uniformly dispersed in solid solution. Copper is an example; it gives the oxide

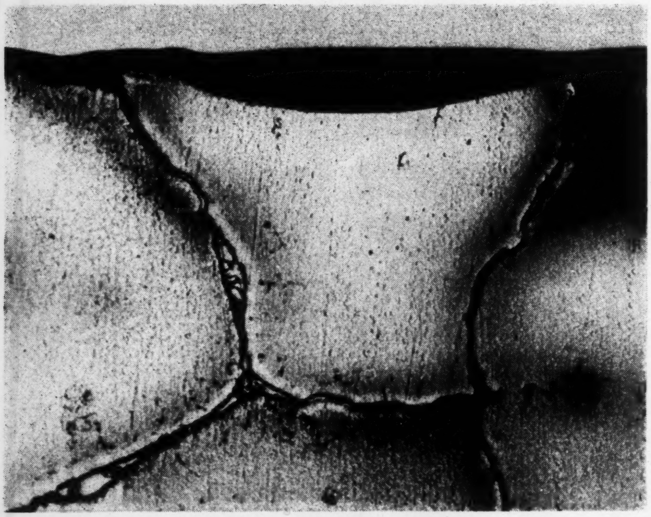


FIG. 14.—The Rough and Irregular Oxide Coating on an "As Cast" 195 Alloy (Aluminum-Copper) Sample (× 500).

The sulfuric acid electrolyte attacked the $CuAl_2$ network preferentially and produced an oxide coating of varying thickness because of solid solution coring within the grains. Vertical illumination. Keller's etch.

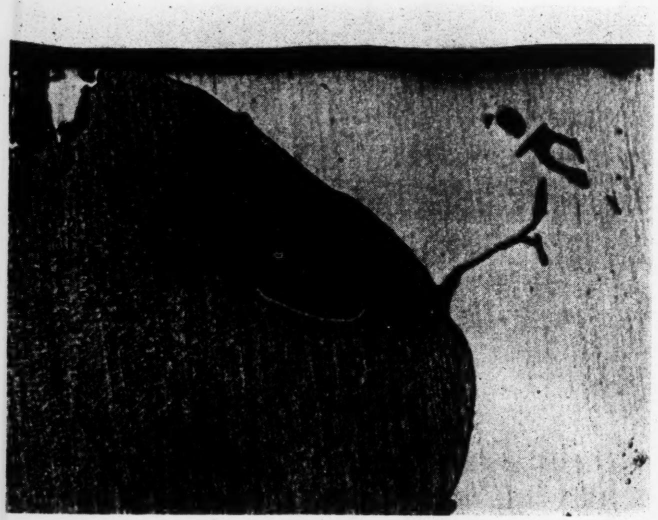


FIG. 15.—The Smooth Uniform Oxide Coating on the Same Alloy as in Fig. 14 when the Sample Was Given a Solution Heat Treatment Prior to the Anodic Treatment (× 500).

This heat treatment dissolved the $CuAl_2$ and eliminated the coring. Reflected light. Vertical illumination. Keller's etch.

coating a yellow color when present in certain amounts. Other elements, such as silicon and manganese, when they are not in solid solution, are responsible for the formation of dark and dull oxide coatings. Thus, it is not uncommon for different tempers of the same alloy to have a different appearance after an anodic treatment. An example of this difference in appearance is shown by Figs. 12 and 13.

Thermal Treatment:

In general, however, thermal treatments which dissolve the alloying constituents and retain them uniformly distributed in solid solution tend to give a metal which takes a more uniform and better appearing oxide coating. Examples of the effect of heat treatment are shown by Figs. 14 and 15. The first illustration shows a cross-section of an anodically coated No. 195 alloy (4 per cent copper) sand casting in the cast condition. In this condition, the alloy contains a network of $CuAl_2$ and solid solution coring along the grain boundaries. The oxide coating on the sample shows the effect of these variations in structure. The coating is rough and oxidation has occurred along the $CuAl_2$ network at the grain boundaries. Since $CuAl_2$ is one of the constituents that is oxidized or dissolved completely at a more rapid rate than the aluminum matrix, the network in this alloy oxidizes more rapidly and in preference to the matrix. Also the difference in the oxidation characteristics of the cored and noncored zones of the grains is partly responsible for the nonuniform oxide coating on the matrix of the grains.

The oxidation characteristics of this cast alloy can be changed, however, by thermal treatment. After a solution heat treatment, these characteristics are greatly improved as is shown by Fig. 15. This treatment causes solution of the $CuAl_2$ network and distributes the copper uniformly throughout the grains. With such a structure, oxidation proceeds uniformly and a more satisfactory oxide coating is obtained. Similar oxidation characteristics are obtained on aluminum-magnesium casting alloys (7) in the cast and heat-treated conditions. The Al-Mg constituent, similar to $CuAl_2$, is rapidly oxidized or dissolved during the oxidation treatment.

Influence of Original Ingot Structure:

Since the anodic oxidation treatment reveals features which can be traced to the original cast structure, the cause of variations in surface appearance must be related to the original ingot structure. Aluminum alloy ingots, in general, do not have an entirely uniform microstructure because of selective solidification and of variations in rate of solidification. During the freezing of an ingot, a dendritic structure is formed wherein the grain boundaries and areas within the grains contain a network of eutectic composition and are bordered by a narrow zone which is rich in the alloying elements.

In view of these structural conditions, it is to be expected that different regions of the cast grains would have different oxidation characteristics. The important point in connection with ingot structure, however, is that variations in ingot structure are sometimes carried through to the finished product in spite of the various fabrication procedures employed. The constituent pattern, however, is elongated into a banded structure by extrusion, rolling or other directional working operations. Thus, the constituent pattern is changed both by the type and the amount of working. From this, it follows that the tendency for anodically treated metal to develop streaks and discoloration is related to the types of constituents present and to the size, arrangement and oxidation characteristics of these constituents.

Conclusions:

It is considered that microscopic examination of anodically coated aluminum alloys by the methods that have been described provides a useful means for determining the characteristics of oxide coatings in general and the variations in the appearance of different alloys treated by the various oxidation treatments. The illustrations serve to show the relationship between the microstructural features of aluminum alloys and the characteristics of the oxide coatings produced by anodic oxidation treatments.

Acknowledgment:

The author acknowledges the skillful work of G. W. Wilcox in the development of the technique for preparing the thin sections of oxide coatings and in the preparation of the photomicrographs.

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THICKNESS OF ANODIC COATINGS ON ALUMINUM

BY JUNIUS D. EDWARDS[1]

SYNOPSIS

The thickness of an oxide coating is a characteristic which may help determine its usefulness for a variety of purposes. The measurement of coating thickness therefore becomes a matter of commercial importance. This article presents some of the methods which may be employed for measuring the thickness of an oxide coating, and gives information regarding the accuracy and field of application of these methods.

Aluminum exposed to the atmosphere always bears on its surface a natural film of oxide which is protective in character. This film is very thin, and various estimates ascribe to it a thickness of the order of 0.5×10^{-6} in. (**10, 11**).[2] By certain anodic treatment processes, oxide films of greater thickness are readily produced on aluminum. A thin and impervious oxide film is produced by anodic treatment of aluminum in an aqueous solution of boric acid. This is the type of oxide film which serves as a dielectric in the electrolytic condenser. These films generally show interference colors. Their thickness will vary with the forming voltage; films (**9**) formed at 220 v., for example, were found to have a thickness of about 10×10^{-6} in.

Another class of oxide films formed anodically on aluminum is characterized by a substantial thickness and a pervious or porous structure. These films are produced by anodic oxidation in electrolytes such as solutions of sulfuric, chromic, or oxalic acids. Within quite a range, the thickness of these oxide films is only limited by the amount of current employed in their production. They have a variety of useful properties, such as resisting abrasion and protecting against corrosion. The extent of the protection which the oxide offers may depend in substantial measure upon the thickness of the coating. The measurement of film thickness has therefore become an important means of appraising the quality of these coatings.

In a sulfuric acid electrolyte, such as is employed in the Alumilite process,[3] a coating of 0.0004-in. thickness is formed by ½-hr. anodic oxidation at a current density of 12 amp. per sq. ft. (**3**). Generally speaking, the Alumilite coatings with the widest use have a thickness of about 0.0001 to 0.0006 in. The coatings formed in chromic acid electrolyte are somewhat thinner, as a rule, and have a thickness on the order of 0.0001 in. Coatings such as these are therefore

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 965.

[3] Protected by patents owned by the Aluminum Company of America.

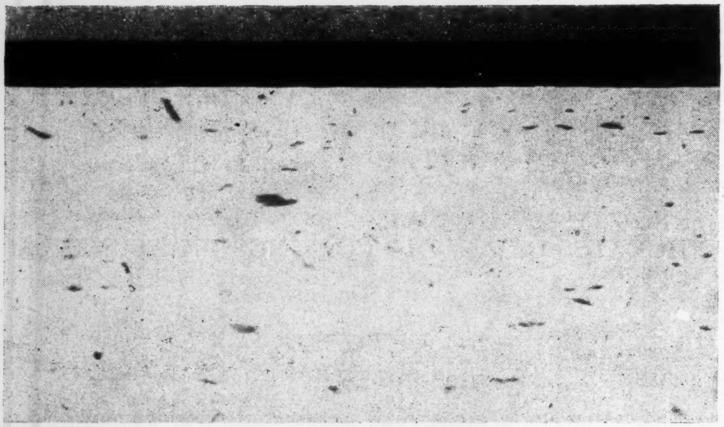

FIG. 1.—Anodic Oxide Coating on Commercial Aluminum Sheet (2S) Produced in Sulfuric Acid Electrolyte, Unetched (×500).

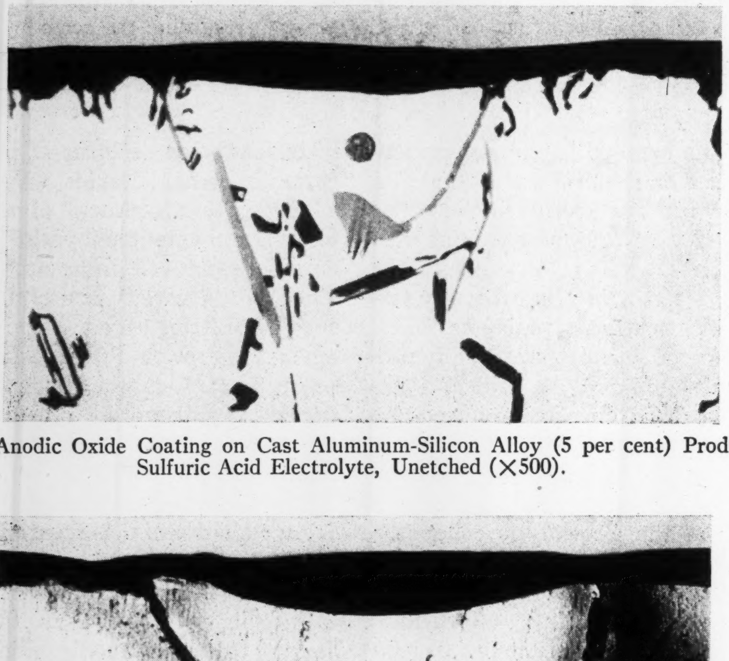

FIG. 2.—Anodic Oxide Coating on Cast Aluminum-Silicon Alloy (5 per cent) Produced in Sulfuric Acid Electrolyte, Unetched (×500).

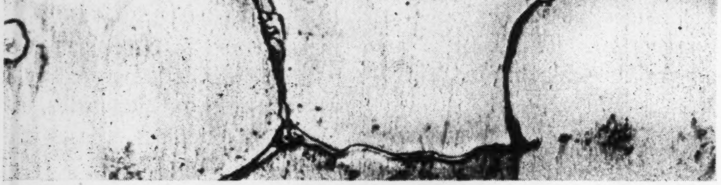

FIG. 3.—Anodic Oxide Coating on Cast Aluminum-Copper Alloy (4 per cent) Produced in Sulfuric Acid Electrolyte, Unetched (×500).

about one thousand times thicker than the oxide film naturally forming on aluminum, and would be expected to have quite different properties, particularly as regards the protection they offer against abrasion and corrosion.

A particular characteristic of these thicker oxide coatings is their porous structure. For the oxide to continue to grow in thickness during the anodic treatment, it is necessary that the electric current continue to flow to the metal surface. The oxide itself is a nonconductor, and there must be innumerable capillary pores or channels which penetrate the coating and carry the electrolyte. The myriads of pores in the coating have a very extensive surface and make the oxide film highly adsorptive. This power to adsorb certain substances gives the film the property of being colored by dyes and mineral pigments.

It is a curious fact that the oxide coating, being formed from the base aluminum instead of being deposited from the solution, grows in the opposite direction to an electroplated coating. The oxide last formed is always between the metal and the oxide previously formed. This means that the oxide first formed has the longest contact with the electrolyte, and where the electrolyte has an appreciable solvent action on the coating—as do the electrolytes last mentioned—it may profoundly influence the properties of the coatings, particularly in the direction of increasing their porosity and decreasing their density. For this reason, no set value can be ascribed to the density of the oxide in these anodic coatings; in fact, it will vary widely, depending upon the electrolyte employed and its temperature, as well as the period of oxidation. The density of the oxide may even vary within the coating, the most porous and less dense oxide being near the surface. It is frequently desirable, therefore, to know both the thickness of the oxide coating and its weight or average density.

Measurement of Coating Thickness with the Microscope:

For thick oxide coatings, the most direct and accurate method of measuring the thickness is by means of the microscope. A small sample of the oxide-coated metal is first mounted and prepared in the manner usually employed for the metallographic examination of metals. In mounting, a procedure must be used that will protect the edges of the specimen against damage during the cross-sectioning and polishing operations. The details of the mounting procedure have already been described (4). With the sample properly mounted and polished, the thickness of the oxide cross-section can be measured directly with a microscope equipped with a calibrated micrometer eyepiece. If a micrometer eyepiece is not available, the image of the cross-section can be thrown on a screen at known magnification and the magnified thickness of the coating measured with a rule.

If the article under examination is a wrought product, the coating will probably be quite uniform in thickness as shown, for example, by the oxide coating on commercial aluminum sheet (see Fig. 1). It would not be difficult to decide how many readings to take to obtain a fair average for such a test specimen. If, however, the sample under examination is from a casting, the coating will probably show more variation in thickness from place to place and there may be some question as to the number of observations necessary in order to obtain what might be called an average measurement (6, 7). In some cases there may be sufficient variation in thickness so that maximum, and particularly minimum, values should be recorded. No established rules can be laid down, however, and each case must be considered on its merits. Oxide coatings on two different commercial casting alloys are shown in Figs. 2 and 3.

If the oxide coating is sufficiently transparent and a petrographic microscope with a calibrated vernier fine adjustment is available, an approximate thickness measurement can be made by the "displacement of image" method. In this method, the microscope is focused first on the interface between the metal and the oxide, and then focused on the top surface of the oxide. A pencil mark on the oxide aids in focusing on the surface, but the adjustment is difficult at best. The difference between the two readings on the fine adjustment, multiplied by the refractive index of the oxide, gives directly the thickness of the coating. According to Chamot, the thickness thus found with an ordinary microscope is accurate to within about 0.02 mm., which is scarcely sufficient for coatings of the kind under consideration (2). Käpernick, however, claims to have secured reasonably satisfactory measurements on oxide coatings by this method (5).

The refractive index of the oxide has been found to vary with its method of production and method of sealing. Films on aluminum sheet of high purity, produced by oxidation in sulfuric acid electrolyte, have shown a refractive index (3) of 1.59 which, after sealing in hot water, was increased to 1.62.

Coatings Showing Interference Colors:

Thin oxide coatings may show interference colors. Coatings made in boric acid electrolyte for electrolytic condensers usually exhibit this phenomenon. It is possible to measure the thickness of this type of coating by the method of Blodgett and Langmuir (1), using as color standards a series of known layers of barium stearate on polished chromium. However, the oxide coatings employed for their decorative and protective effect are generally too thick to be measured by this method, so no attempt will be made to give the details of procedure, and the reader is referred to the original article. Still other optical methods are available for estimating the thickness of films which show interference colors (8).

Measurement of Coating Thickness by Stripping Method:

Another class of methods depend on measurements of the article before and after removal of the oxide coating. This measurement may be either a direct thickness measurement or may be a weighing of the article before and after removing the oxide coating, in which case the determination yields the weight of oxide on a given area of the article. In the 1937 report of A.S.T.M. Committee B-7, details are given for a stripping method which employed a solution of sulfuric acid to dissolve the oxide (4). The sulfuric acid solution contained a small amount of antimony to serve as an indicator; when the oxide had dissolved to the point that bare aluminum was exposed, antimony deposited as a gray to black film and indicated that the stripping operation was complete. The solution, however, was not particularly selective and attacked the metallic aluminum as soon as it was exposed. It is necessary by this method to take great care to stop the process at the point where all the oxide has been removed. Since then, the process has been improved by Ralph B. Mason of Aluminum Research Laboratories. The new method depends upon the use of a solution containing chromic and phosphoric acids for removal of the oxide. Instructions for carrying out the method are as follows:

Determination of the Weight of Coating Reagents:

Phosphoric acid, 85 per cent	35 ml.
Chromic acid, CrO_3	20 g.
Water to make	1000 ml.

For the determination of weight of coating, use a sample of known surface area; about 9 sq. in. is usually a convenient size. If the oxide coating is contaminated or impregnated with oil, grease, lacquer, etc., it may be desired to remove as much as possible of this material before determining the weight of coating. It is not usually practical to remove these materials from the pores of the coating but surface films can frequently be removed by wiping the sample with a cloth wet with a suitable solvent.

Weigh the sample and then immerse in the phosphoric-chromic acid solution for 5 min. at 212 F., to remove the oxide whose weight is to be determined. Remove the sample, wash in distilled water, dry and weigh. Repeat the 5-min. exposure until the coating is completely removed, which is indicated by the sample's weight remaining constant.

Report the weight of coating in milligrams per square inch of coating.

Notes:

The thickness of the oxide film can also be determined by measuring the thickness of the sample with a micrometer before and after removing the oxide.

The rate at which aluminum oxide dissolves in the phosphoric-chromic acid solution decreases as more and more oxide is dissolved. The solution should be discarded after about 1 liter of solution has dissolved 5 g. of aluminum oxide. The addition of 21 ml. of 85 per cent H_3PO_4 to the solution containing 5 g. Al_2O_3 per liter will be equivalent to preparing a fresh solution.

If coating is to be determined only on one side, take a sample which is somewhat greater than required for final size and remove coating from other side by swabbing with a hot solution of caustic soda (10 per cent NaOH). Before such swabbing, outline an area somewhat greater than 3 in. square with paraffin; this will prevent caustic soda running over edges and attacking the coating on the other side. Swab until bare metal is exposed, which is indicated by uniform bubbling over the entire surface. Swab with hot distilled water and dry with cloth. Swab with 1:1 HNO_3 solution. Rinse with hot distilled water and dry at 212 F. Cool and shear to desired size, preferably 3 in. square.

The method gives readily reproducible results, as will be seen by comparison of the data in Table I, which show the measurements of the weight of oxide on a series of samples numbered *A* to *H*, as determined in three different laboratories.

TABLE I.—MEASUREMENT OF WEIGHT OF OXIDE FILM ON ALUMINUM SHEET.

Sample	Weight of Oxide Coating, mg. per sq. in. of coating			Average Thickness of Oxide Coating Measured Under the Microscope, in.	Density, g. per cu.cm.
	Aluminum Research Laboratories	Bell Telephone Laboratories	Bohn Aluminum and Brass Corp.		
A	8.0	8.5	7.8	0.00015	3.25
B	14.4	15.6	13.9	0.00029	3.03
C	21.1	22.8	20.2	0.00044	2.93
D	28.9	31.5	27.4	0.00058	3.04
E	7.3	7.1	7.7	0.00016	2.79
F	15.7	16.7	14.9	0.00030	3.19
G	21.8	23.2	21.3	0.00044	3.02
H	29.9	31.2	28.7	0.00058	3.14

The density values for the coatings of Table I are in the neighborhood of 3, while massive crystalline alumina has a density of about 3.9 to 4.0.

Thickness of Coating versus Dielectric Strength:

Oxide coatings produced by anodic treatment of aluminum may have a substantial dielectric strength and, in general, this dielectric strength increases linearly with thickness of coating. The breakdown voltage of the coating may therefore be used as a rough measure of the thickness of the coating. There are, however, quite a number of variables other than thickness which affect the breakdown voltage of the coating. The dielectric strength may vary, for example, with the alloy being coated, with the electrolyte employed and its temperature, and the current density, as well as the method used for sealing the coating. Voltage breakdown values vary also with the character of the surface preparation; coatings on a sand-blasted surface, for example, have many points which show a low breakdown voltage.

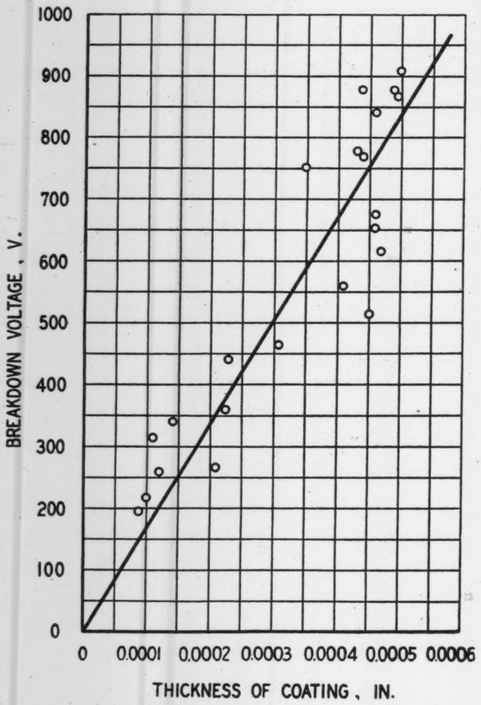

FIG. 4.—Relation Between Breakdown Voltage and Thickness of Sealed Oxide Coating on Commercial Sheet Aluminum.

It is not practical, therefore, to attempt to determine the thickness of a coating of unknown characteristics by a measurement of its breakdown voltage. However, where the characteristics of the coating are known, as in the case of routine production, the breakdown voltage can serve as a quick test of the approximate thickness of the coating, in order to make sure that the coating is within established limits. The minimum breakdown voltage is determined empirically by a series of measurements on coatings which have been found to be satisfactory by other tests.

In Fig. 4 are shown the voltage breakdown values for approximately 22 different samples of commercial sheet aluminum bearing coatings up to about 0.0005 in. in thickness. Some of these coatings were made in different plants and under different coating conditions; all of them were sealed by treatment with hot water. The maximum variation for coatings of the same thickness is not great and, in fact, the agreement was somewhat better than has been obtained in other similar series of measurements. These measurements were carried out by the method outlined in A.S.T.M. Tentative Method of Test for Dielectric Strength of Anodized Aluminum (B 110 - 39 T).[4]

In Closing:

Some of the methods described in this paper for determining the thickness of oxide coatings on aluminum give precise values and are best adapted for laboratory utilization; others give approximate values and are satisfactory for the control of plant production. The fact should not be overlooked that even simpler tests are available which may yield considerable information in the hands of an experienced person. A knife or scribe, for example, will usually disclose both by the depth of scratch and the sound, as it cuts through the oxide, whether the coating is thick or thin, and tell something about its hardness. In the case of very thin coatings, an ink eraser, or even a pencil eraser, may cut through the oxide in a relatively few strokes and give reliable qualitative information.

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[4] 1939 Book of A.S.T.M. Standards, Part I, p. 1040.

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DISCUSSION

Mr. Sam Tour.[1]—Mr. Edwards, in explaining the formation of anodic coatings, showed how the coating is attacked by the electrolyte at the surface and the metal is transformed into the coating as the oxidizing action penetrated. I wonder whether Mr. Edwards could enlarge a bit on this. He further stated that in the Alumilite coating process, a coating of 0.0004 in. thick is commonly produced, while in the normal chromic acid process, the coating is about 0.0001 in. How much metal has been lost on the surface in each case? We all know that aluminum oxide or hydrated aluminum oxide or whatever it may be has a larger volume than aluminum metal. Often the assumption is made that in anodizing a piece of aluminum there is an increase in size—how much is that increase in total size and how much actual decrease in metal size is there?

Mr. Junius D. Edwards.[2]—In some experiments we performed some time ago, we took a piece of aluminum foil and oxidized it step by step by the Alumilite process, and at each successive step its thickness increased. When we had oxidized it completely through, as we did, there was an increase of about thirty per cent in the thickness of that foil. In commercial operation you may experience an increase in thickness of the part, no increase in thickness, or sometimes an actual decrease in thickness of the part, depending upon the electrolyte employed, the temperature of the electrolyte, and the time it remained immersed in the electrolyte. The conditions chosen for the experiment just described were such that there was a minimum of solvent action on the coating because we wished to ascertain what the maximum increase of thickness would be in transforming aluminum into aluminum oxide.

Mr. Tour.—That would indicate that for a 0.0004-in. coating, the maximum increase in over-all dimensions would not be much more than a tenth of a thousandth, or about a third of the coating thickness?

Mr. Edwards.—That is right. It should be remembered, however, that usually the part is oxide-coated all over and two thicknesses of coating are involved in measuring the diameter or thickness of a part.

Mr. Tour.—I am under the impression that in many commercial applications allowances for increase in size of as much as half of a thousandth are being made.

Mr. Edwards.—I cannot understand on what basis they would make that allowance.

Since this discussion took place, one aluminum and two aluminum alloy cylinders, 0.6 in. in diameter, were measured before and after anodically coating in chromic acid electrolyte. The coatings were approximately 0.0001 in. in thickness and the average increase in diameter as a result of the anodic treatment was 0.00001 in. with a maximum observation of 0.00008 in.

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[2] Assistant Director of Research, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

THE ABRASION RESISTANCE OF ANODICALLY OXIDIZED COATINGS ON ALUMINUM

By H. G. Arlt[1]

Synopsis

The development of an accurate air-blast abrasion testing apparatus and technique is described. This test is suitable for the determination of the abrasion resistance of oxide coatings on aluminum and of other organic and inorganic coatings. The test equipment consists of a calibrated glass nozzle in which the abrasive is mixed with compressed air and which is housed in a suitable container arranged for the easy insertion and observation of the specimens under test. Information is provided as to the necessary control apparatus to insure a uniform supply of low pressure air at closely controlled pressure and rate of flow. Data are included on several sets of anodically oxidized coatings on aluminum to show the degree of duplicability of results both in a single laboratory and in several cooperating laboratories. Additional abrasion resistance data on anodically oxidized coatings on aluminum are included and are related to the thickness, oxidizing time and sealing conditions in order to illustrate the value of abrasion resistance information on coatings of this character. Abrasion data are useful not only in the development of oxidized coatings but also in directly determining the useful life of such finishes under abrasive environmental conditions.

The extensive use in industry of oxide coatings on aluminum focuses attention on the necessity for suitable methods of test to determine and control the useful properties of these coatings. The research, design, and manufacturing problems involved can be greatly facilitated by reliable test methods for those characteristics that are definitely related to the methods of coating as well as to the use of anodically oxidized aluminum, that is, such properties as corrosion resistance, thickness, abrasion resistance, hardness and electrical breakdown. Much has been accomplished toward this end, a method for electrical breakdown having already been approved as tentative by the Society,[2] while methods of test for other properties are rapidly being developed by several investigators.

It is the purpose of this paper to discuss the measurement of abrasion resistance of oxide coatings on aluminum by abrasive air blast and to present data obtained by this method to demonstrate their degree of duplicability and usefulness.

The various methods of measurement of abrasion resistance may in general be grouped under two headings, (1) those

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[1] Member of Technical Staff, Bell Telephone Laboratories, New York, N. Y.

[2] Tentative Method of Test for Dielectric Strength of Anodized Aluminum, (B 110 - 39 T), 1939 Book of A.S.T.M. Standards, Part I, p. 1040.

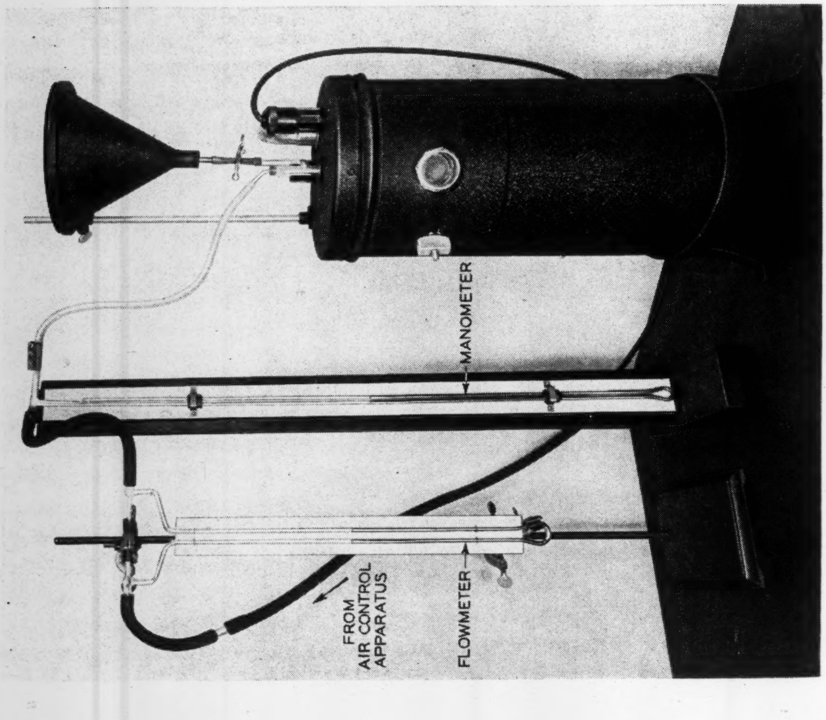
FROM AIR CONTROL APPARATUS
FLOWMETER
MANOMETER

FIG. 1.—Abrasion Test Apparatus (Closed).

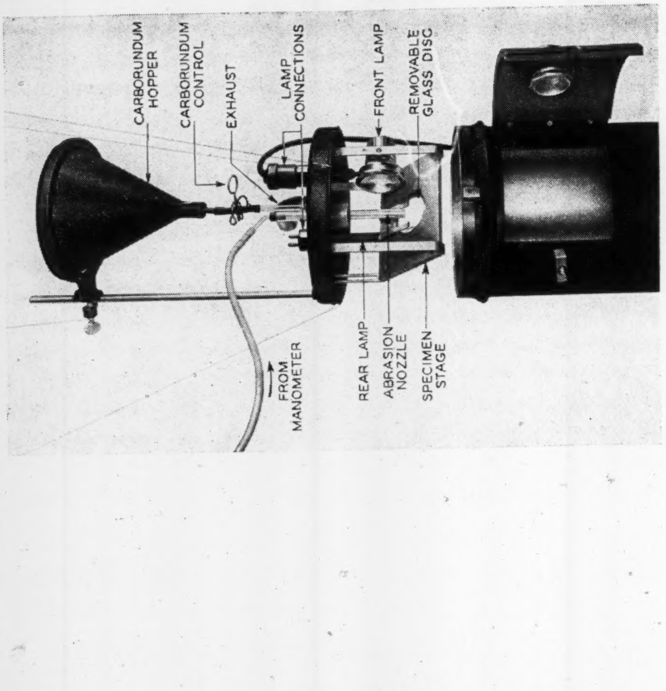
CARBORUNDUM HOPPER
CARBORUNDUM CONTROL
EXHAUST
LAMP CONNECTIONS
FRONT LAMP
REMOVABLE GLASS DISC
REAR LAMP
ABRASION NOZZLE
SPECIMEN STAGE
FROM MANOMETER

FIG. 2.—Abrasion Test Apparatus (Open).

which employ a finely divided abrasive which is caused to impinge on the surface tested, and (2) those which involve the rubbing, sliding or rolling action of two surfaces. Although methods in the second group are very useful in solving specific practical problems, particularly where the mechanical motions employed are directly related to usage, the degree of duplicability for general investigational purposes does not appear to be as good as when using methods of the first type. This is undoubtedly due to the difficulties of controlling (*a*) the heat developed by wear under the changing condition of the wearing surfaces during testing, (*b*) the dimensional relations, (*c*) the mechanical motions, and (*d*) the pressure of contact. Some preliminary work has been carried out with a device of the rolling and sliding type but the results on duplicate specimens were in such poor agreement that the method was abandoned.

The abrasive air-blast test measures the cohesion of the coating and is also related to the hardness and density as well. A means of accurately measuring abrasion resistance is therefore of assistance in the study of the effect of the oxidizing and sealing conditions on the properties of the coatings. The close similarity between the abrasive air-blast test and the abrasive conditions met with in service on such equipment as trains, automobiles, and airplanes also makes this test useful in selecting finishes of maximum abrasion resistance for such uses.

APPARATUS

Of the several variations in use of the abrasion by impingement method, that of Schuh and Kern[3] was selected for measurement of the abrasion resistance of anodically oxidized coatings in view of its success as a method of measuring the same property of organic protective and decorative coatings.

The apparatus employed is illustrated in Figs. 1, 2, 3, and 4. The abrasion device consists essentially of a glass nozzle in which the abrasive is mixed with air at controlled pressure and impinged on the surface of the specimen under test.

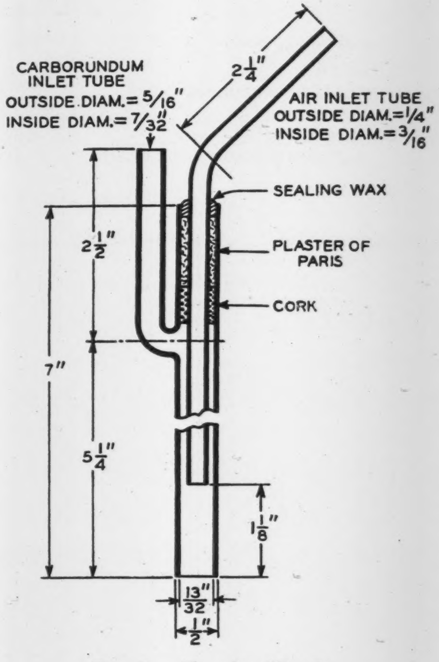

FIG. 3.—Abrasion Nozzle.

The nozzle of Fig. 3 was employed to obtain most of the data reported. Some of the data was obtained on an earlier design of nozzle in which the dimension from the abrasive inlet to the outlet was 2.4 in. instead of $5\frac{1}{4}$ in. as shown on Fig. 3. The entire device is housed in a container to catch the used abrasive and is normally arranged for testing specimens up to 3 by 3 by $\frac{1}{4}$ in. It is constructed with all the essential equipment mounted on the top of the container so that the coatings on larger specimens or full-size parts can be tested by removing the top assembly and placing it directly on the part to be tested.

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[3] A. E. Schuh and E. W. Kern, "Measurement of Abrasion Resistance," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 3, pp. 72–76 (1931).

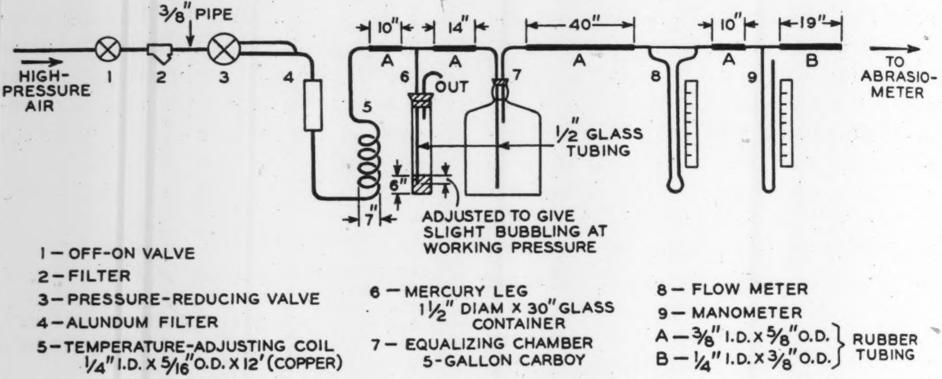
3/8" PIPE
HIGH-PRESSURE AIR
1
2
3
4
5
6
OUT
7
8
9
10"
14"
40"
10"
19"
A
A
A
A
B
TO ABRASIOMETER
1/2" GLASS TUBING
6"
7"
ADJUSTED TO GIVE SLIGHT BUBBLING AT WORKING PRESSURE
1 – OFF-ON VALVE
2 – FILTER
3 – PRESSURE-REDUCING VALVE
4 – ALUNDUM FILTER
5 – TEMPERATURE-ADJUSTING COIL 1/4" I.D. X 5/16" O.D. X 12' (COPPER)
6 – MERCURY LEG 1 1/2" DIAM X 30" GLASS CONTAINER
7 – EQUALIZING CHAMBER 5-GALLON CARBOY
8 – FLOW METER
9 – MANOMETER
A – 3/8" I.D. X 5/8" O.D.
B – 1/4" I.D. X 3/8" O.D.
RUBBER TUBING

Fig. 4.—Air Control for Abrasion Test.

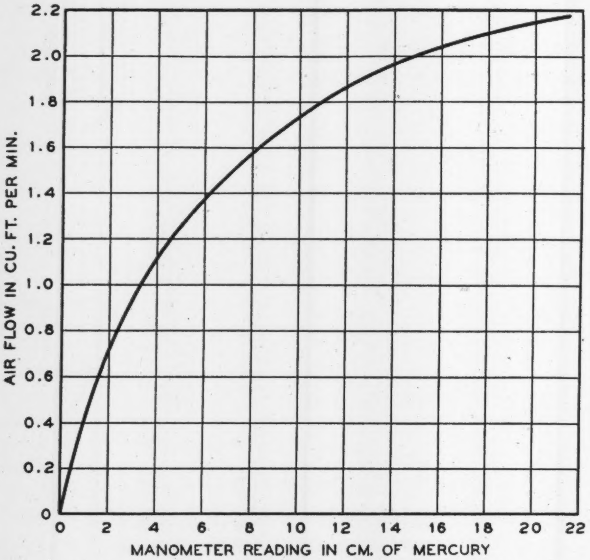

Fig. 5.—Air Pressure *versus* Air Flow – Abrasion Test Apparatus.

The axis of the nozzle is fixed at 45 deg. to the surface of the specimen and is arranged in a bushing so that it can be slid downward into contact with the surface of the specimen. The angle of incidence of the nozzle and the distance between nozzle and specimen are arbitrary, and were selected for convenience. An increase in the angle of incidence increases the abrasion value and an increase in the distance between the nozzle and specimen also increases the abrasion value as shown by Schuh and Kern.

On the cover of the test machine are mounted the nozzle, exhaust air vent, specimen holder, lights for illumination of the specimen under test and a support for the abrasive container. The housing is equipped with a hinged door for insertion and removal of specimens. This door also contains a window for observation of the specimen during test. The cover is equipped with an exhaust air vent to eliminate back pressure in the test chamber.

Figure 4, which is a schematic drawing of the details of the air control, shows an arrangement which is suitable for a high-pressure air line but can readily be adapted for an individual compressor. In order to obtain results of good duplicability the air pressure as measured on the mercury manometer next to the nozzle was controlled so that it did not vary more than ± 1.5 mm. of mercury from the nominal value during a test. This was accomplished by use of a special design of diaphragm-type reducing valve. On the low-pressure side, a difference of 3 mm. of mercury pressure corresponds to an air flow of about 0.03 cu. ft. per min. at 10 cm. of mercury, the test pressure usually employed. The relation between pressure and air flow in the equipment described is shown on Fig. 5. The size or length of connection from manometer to nozzle does not appear to be particularly critical. The $\frac{1}{4}$-in. inside diameter rubber tube connection used was 20 in. long. A decrease of 30 per cent in length did not effect a flow difference which could be detected on the flow meter. An increase in inside diameter of this tube from $\frac{1}{4}$ in. to $\frac{3}{8}$ in. increased the flow by only 0.02 to 0.03 cu. ft. per min. The other connections are not critical provided that adequate size is employed (at least $\frac{3}{8}$ in. in inside diameter).

TABLE I.—ABRASION VALUES OBTAINED BY DIFFERENT LABORATORIES.

Sample	Alloy	Oxidizing Time, min.	Thickness, in.	Abrasion Values, g. of carborundum												
				Laboratory 1			Laboratory 2				Laboratory 3					
				Readings		Average	Readings			Average	Readings					Average
A	2S-H	10	0.00015	30	26	28	30	32	34	32	23	21	24	24		23
B		20	0.00029	86	93	90	81	84		83	59	52	61	61		58
C		30	0.00044	106	104	105	132	132		132	89	89	90	89		89
D		40	0.00058	176	172	174	174	175		175	113	113	117	117		115
E	53-S	10	0.00016	33	32	33	31	32		32	33	36	33	39	39	36
F		20	0.00030	79	77	78	76	82		79	63	67	67	67		66
G		30	0.00044	133	132	133	101	102		102	77	79	78	79		78
H		40	0.00058	200	198	199	181	181		181	128	130	129	132		129

NOTES:

Test equipment of Laboratories 1 and 2 was the same. Laboratory 3 used similar equipment employing a shorter nozzle.

All specimens were anodically oxidized under the same conditions and were unsealed. The specimens were made at the Research Laboratories of the Aluminum Company of America.

TESTS

Eight large aluminum sheets were anodically oxidized by the commercially used Alumilite process[4] under identical conditions except for different periods of time. Four sheets were of 2S-H alloy and four were of 53-S alloy. Oxidizing times of 10, 20, 30, and 40 min. were employed. The large sheets were cut up after coating and duplicate specimens were distributed to three laboratories for preliminary tests. Two of the laboratories had the testing equipment illustrated herein except for the pressure control valve and flow meter. The nozzles of these two machines were not calibrated against each other. The third laboratory used essentially the same equipment as the other two laboratories except for the shorter nozzle described above.

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[4] Protected by patents owned by the Aluminum Company of America.

Abrasion values were obtained by the three laboratories at 10 cm. of mercury air pressure. The environment at time of test was not controlled at the three laboratories conducting the tests and the degree to which the variation in air pressure during each test was controlled is not known. The end point of each determination was, by agreement, the removal of the oxide coating from an area 2 mm. in diameter. The thickness of each coating was determined by measurement on a microscope of a sectioned and polished specimen imbedded in a transparent plastic. The data obtained are given in Table I.

An examination of the data given in Table I shows that the abrasion values from the three participating laboratories are consistent in their evaluation of abrasion resistance of the coatings tested even though the actual values are numerically different. The abrasion resistance of the oxide coatings appears to increase with thickness in essentially the same fashion when determined by any one of the three cooperators.

It should be noted that the data of laboratories 1 and 2 are in reasonable numerical agreement even though the nozzles were not calibrated against each other. The data of laboratory 3 cannot be expected to be in numerical agreement with laboratories 1 and 2 due to appreciably different nozzle dimensions.

In considering these data, it should be kept in mind that the thickness determinations were not made at the same areas as the abrasion determinations. There are indications from other data on the same specimens of small variations in thickness of the oxide coating over each large sheet. If this is so, the abrasion values, which vary with thickness, cannot be uniform all over a sheet.

The abrasion values reported were obtained in some cases on both sides of the coated sheets and this may also have had an effect on the data.

In order to ascertain the reproducibility of abrasion values in one laboratory, comparative tests were made 3 yr. apart on the same test equipment by different operators, on the same specimens. The averages of the results are given in Table II. Unfortunately, the nozzle used for the first determinations had been broken and replaced by a duplicate. In such cases it has been customary to establish a calibration factor, comparing abrasion values for the two nozzles on the same specimen under the same operating conditions. Experience has shown this to be a safe procedure for nozzles that are essentially the same except for slight variations in glass tube size and in precision of blowing and adjustment of the air jet in the outer tube. This procedure would be questionable if used to calibrate nozzles of appreciably different dimensions.

TABLE II.—TOTAL ABRASION RESISTANCE, AVERAGE VALUES.

Sample	1936	1939 Actual	Corrected (× 1.18)
A	27	23	27
B	70	58	69
C	100	89	105
D	130	115	136
E	41	36	43
F	84	66	78
G	111	78	92
H	154	129	152

NOTES:

1939 readings made by a different operator than the 1936 readings.

A different nozzle was used in 1939 than in 1936. A calibration constant of 1.18 was used to permit comparison of values obtained with the two nozzles.

Each 1936 value is the average of 12 measurements. Each 1939 value is the average of 4 measurements.

The data of Tables I and II show that the standardization and control of the test equipment and methods of operation require improvement before the method can be used safely for inspection purposes.

The use of a high-pressure air line as

a supply for abrasion testing resulted in pressure variations during test due to fluctuations on the high-pressure side caused by other users of the line. This required manual control of the reducing valve during testing to maintain a reasonably uniform low-pressure air. Even with careful manual control, the pressure fluctuated as much as ± 1 cm. of mercury momentarily during a test period. In order to correct this condition as far as possible, a special type of diaphragm reducing valve with supplementary bleeder control was installed. At the same time a flow meter was introduced before the manometer as shown on Fig. 4 so that duplicate equipment could be adjusted to give the same air flow at the same pressure.

A further set of data was obtained by one laboratory with the improved control of air pressure and flow. These data, see Table III, were obtained on four sets of Alumilite process coated specimens representing different oxidizing and sealing conditions. An air pressure of 12 cm. of mercury was employed which corresponds to an air flow of 1.8 cu. ft. per min. The weight of 180 mesh carborundum required to wear through the oxide film was determined under these conditions. The end point employed was an area of approximately 2 mm. in diameter of exposed base metal. When in doubt, the exposure of base metal was checked with a metallic feeler connected to the base metal with a battery and head set in series. All these abrasion measurements were made at 50 per cent relative humidity at 77 F. after the specimens had been equilibrated to these conditions.

The measurements were made on one side of each specimen and the thicknesses were determined by microscopic cross-section on the same side. In general,

TABLE III.—ABRASION RESISTANCE OF ANODICALLY OXIDIZED COATINGS ON ALUMINUM ALLOY 2S-½H.

H_2SO_4 Bath—10 per cent concentration, 95 F.

Panel Code	Oxidizing Time, min.	Sealing		Current Density, amp. per sq. ft.	Thickness of Oxide, in.[c]	Abrasion Resistance					
						Total, g.					g. per 0.001 in.
		Solution	Time, min.			Readings				Average	
1W	10	Water[a]	10	14.4	0.00021	27	27			27	129
2W	20		20		0.00037	42	40			41	111
3W	30		30		0.00056	53	53			53	95
4W	40		30		0.00061	60	56			58	95
5W	50		30		0.00065	58	57			58	89
6W	60		30		0.00090	78	76			77	86
1U	10	None			0.00016	34	32			33	206
2U	20				0.00030	86	86			86	286
3U	30				0.00046	170	170			170	369
4U	40				0.00060	237	234			236	394
5U	50				0.00054	233	216	219		223	413
6U	60				0.00055	210	208			209	398
1D	10	Sodium dichromate[b]	10		0.00016	25	24			25	156
2D	20		20		0.00033	63	61			62	188
3D	30		30		0.00047	77	78			78	166
4D	40		30		0.00051	91	87	90		89	174
5D	50		30		0.00052	96	94			95	183
6D	60		30		0.00055	98	103	102		101	184
1Y	30	Water[a]	30	5	0.00006	7	7			7	117
2Y				10	0.00033	27	27			27	82
3Y				20	0.00073	76	80	75		77	105
4Y				25	0.00098	96	101	104	103	101	103
6Y				35	0.00107	119	127	124		123	115

[a] Bath temperature 212 F.

[b] 5 per cent sodium dichromate solution (boiling).

[c] Each thickness value is average of at least 5 measurements.

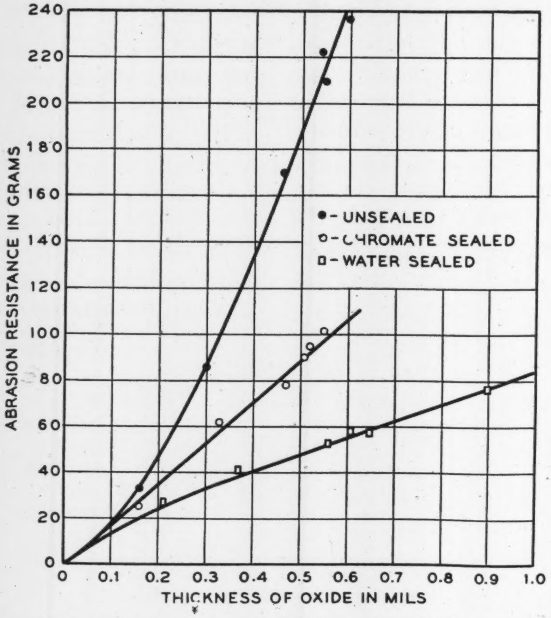

FIG. 6.—Relation of Abrasion Resistance to Thickness for Different Sealing Conditions.

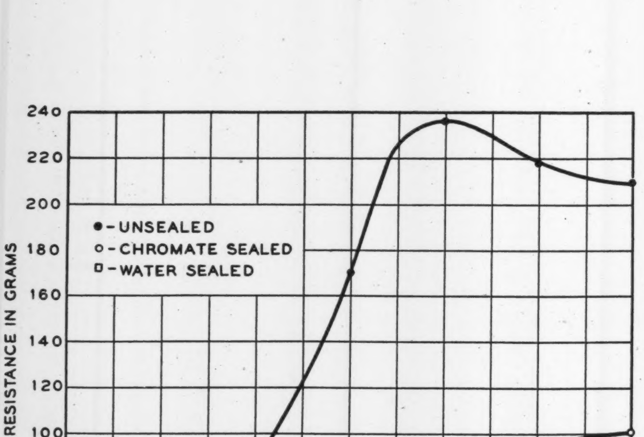

FIG. 7.—Relation of Abrasion Resistance to Oxidizing Time for Different Sealing Conditions.

the thickness determinations were made on areas about 2 or 3 in. from the abrasion spots.

These later data again show an increase of abrasion resistance with thickness and a substantial difference between abrasion values for sealed and unsealed coatings. The values also show considerable improvement in duplicability of values on the same specimen, the largest variation between readings on the same test specimen being 7.3 per cent as compared with 18 per cent in the earlier data. In addition, the number of readings on the same specimen showing appreciable variation has also decreased considerably.

In order to illustrate the usefulness of abrasion resistance data Figs. 6 and 7 have been prepared.

The relation between total abrasion resistance and thickness of the unsealed, water-sealed, and chromate-sealed coatings is shown on Fig. 6. It can be seen from these curves that the unsealed coatings have better abrasion resistance than either chromate or water-sealed coatings, the water-sealed coatings having the lowest abrasion resistance at any thickness level. These curves show that the measurement of abrasion resistance may be related to the degree of hydration of these coatings. The total abrasion resistance of the coatings has been plotted rather than the abrasion resistance per unit thickness since an examination of the data discloses that the average abrasion resistance is not uniform throughout the coatings, particularly in the case of the sealed coatings where the thinner coatings have a higher average abrasion resistance than the thicker coatings. This may be due to the shorter sealing time employed for the thinner coatings and may indicate incompleteness of seal of these coatings.

When the abrasion values are plotted against oxidizing time, see Fig. 7, it appears that the abrasion resistance approaches a maximum for the processing conditions employed. There are some indications that the unsealed coating drops off in abrasion resistance after a critical oxidizing time has been passed. This is also reflected in the thickness values.

CONCLUSION

An air-blast type of abrasion test can be successfully employed to measure the abrasion resistance of anodically oxidized coatings on aluminum. The data obtained are valuable not only with respect to the commercial use of these coatings but for research purposes as well.

The results of this study indicate that careful control of testing conditions produced abrasion data of a high order of precision. Before adopting this method for inspecting product, it would be desirable to conduct a confirmatory cooperative test.

Acknowledgment:

The author hereby acknowledges the helpful assistance of R. J. Phair and A. Mendizza of the Bell Telephone Laboratories, Inc., in this work. Acknowledgment is also made to J. D. Edwards of the Research Laboratories of the Aluminum Company of America and to W. E. McCullough of the Bohn Aluminum and Brass Corp. for the preparation of specimens and cooperative tests.

DISCUSSION

Mr. Sam Tour.[1]—I should like to emphasize that in controlling the air in making an abrasion test, the vital thing is the amount of air, not the pressure. It is not a question of pressure somewhere in the air line back of the instrument; it is a question of the velocity of the air through and from the nozzle. As the size of the nozzle is fixed, the important thing becomes the cubic feet per hour of air that are forced through. An air flowmeter registering in terms of cubic feet per minute or per hour is the important item. In controlling the carborundum side, of course, the important thing is the grain size of the carborundum. Mr. Arlt referred to No. 180 carborundum. I was once under the impression that, when one purchased No. 180 carborundum, practically 75 per cent at least was of the order of 180 mesh with possibly a small amount of 200 or finer mesh, and very little coarser than 150. I find that the so-called No. 180- or No. 170-mesh carborundum available contains considerable dust and will run as much as 50 per cent coarser than No. 150 and with some particles that will not go through a 20-mesh screen. To control the carborundum in these tests, it should be screened and only that fraction between the No. 150 and No. 200 sieves should be used. That means that only about 30 per cent or 25 per cent of the purchased carborundum is usable. In this way a much greater uniformity is obtainable.

Mr. J. D. Edwards.[2]—Mr. Arlt brings out the difference in abrasion resistance between the surface and base of the coating. The significant difference in abrasion resistance resulting from the sealing operation may be related to the changes which take place during the treatment with hot water. The coating, which is initially porous, after being treated with hot water becomes impervious, and this change is accompanied by the conversion of anhydrous aluminum oxide (Al_2O_3) to the hydrated form ($Al_2O_3 \cdot H_2O$). No doubt this chemical reaction between the water and the coating changes the state of organization so that it is less resistant to the abrasive air blast.

Mr. H. G. Arlt[3] (*Author's closure*).—With respect to Mr. Tour's comment involving the control of air pressure and air flow, further study of these variables has been carried on since the work reported on in the paper.

There is very little question but that abrasion values of excellent duplicability can be obtained on any one piece of abrasion test equipment if either the air pressure or air flow is controlled. The two properties are interdependent and vary with respect to the dimensions of the glass nozzle and the length, size, bends, etc., in the connecting tubing. When it is desired to calibrate one test machine against another, it is necessary that both the air pressure and the air flow be controlled on each machine. This can be done by using nozzles of essentially the same dimensions, by the use of an adequate size of connecting tubing and by adjustments in the lengths of tubing and bends. It may be of interest to state that it was found necessary to use a special glass tubing, the inside diameter of which is within plus or minus 0.0004 in. of the specified dimension in order to permit the standardization of several abrasion test machines.

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[1] Vice-President, In Charge of Chemical and Metallurgical Engineering, Lucius Pitkin, Inc., New York, N. Y.

[2] Assistant Director of Research, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[3] Member of Technical Staff, Bell Telephone Laboratories, New York, N. Y.

As Mr. Tour points out, it is essential in this test that the particle size of the abrasive be controlled by adequate means. The abrasive size is, in this test, a measure of the number of particles impinging on the specimen as well as a controlling factor in the cutting energy of each particle. A difference of over 100 per cent in abrasion value can be obtained on the same test specimen when the particle size is varied from 90 to 215 mesh with the air flow and air pressure conditions held constant. The higher abrasion value, of course, is obtained with the coarser abrasive.

ELECTRICAL BREAKDOWN OF ANODICALLY OXIDIZED COATINGS ON ALUMINUM: A MEANS OF CHECKING THICKNESS OF ANODIZED FINISHES

By K. G. Compton[1] and A. Mendizza[1]

Synopsis

The existing methods for determining the thickness of anodically produced oxide coatings on aluminum are relatively few and are almost entirely of a destructive nature. It is a fairly well established fact that, within the thickness limits normally encountered in practice, the voltage breakdown is a linear function of thickness of oxide film. The authors have endeavored to utilize this fact in developing a test method for determining the thickness of coatings produced under known and controlled conditions with practically no injury to the finish. Data are given which show the relationship between breakdown resistance, anodizing time, thickness of coating, current density and sealing of anodically oxidized polished commercially pure aluminum. Statistical data for the values obtained are also given, indicating the good reproducibility of the breakdown values. By calibrating a particular anodic process, satisfactory results may be obtained in a relatively short time and often without destroying or marring the article. Since the oxide coating is not entirely homogeneous it is necessary to obtain a fairly large number of readings for every test condition. The authors have found that approximately twenty-five readings are usually sufficient and can be made in a relatively short time. Although only one of the many anodizing possibilities has been investigated, the applicability of this method of evaluating the thickness of oxide coatings may be extended to all commercial treatments.

An investigation of the voltage breakdown characteristics of anodically produced coatings on commercially pure aluminum has been conducted with a view to determining the effect upon breakdown of such factors as anodizing time, sealing, hardness of base metal, contact pressure, and bending of the oxidized article. It was thought that out of this study might grow a method of checking the thickness of coating on a production basis which would not destroy the sample and at the same time be rapid and simple. It is the purpose of this paper to present the data more from the point of view of establishing a method of evaluating the finish than from that of studying a dielectric.

The need of a rapid method of checking the thickness of this finish has become increasingly more important as the volume of work has increased, particularly in factories employing mass-production methods. Various methods have been proposed, such as the needle penetration test in which a measurement

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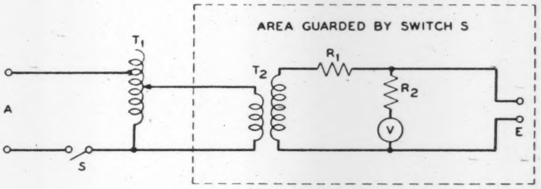
[1] Members of Technical Staff, Bell Telephone Laboratories, New York, N. Y.

is made of the distance the needle travels from the surface of the finish before making an electrical contact to the base metal; stripping the coating from a known area, weighing the specimen and from the loss in weight determining the thickness; grinding a V-shape notch and determining the thickness microscopically; and the standard method of casting a section of the specimen in a plastic, cutting and polishing in a plane normal to the surface of the specimen and measuring the thickness under a microscope. All of these methods suffer either from a lack of precision or from the time and effort required to make the determination. A voltage breakdown method within its limitations would have the advantage of being extremely rapid and frequently could be employed without destroying the sample.



A - LIGHTING CIRCUIT 110 TO 120 V., A C.
S - SAFETY SWITCH OPERATED BY DOOR
T_1 - GENERAL RADIO VARIAC
T_2 - STEP UP TRANSFORMER
R_1 - CURRENT LIMITING RESISTOR
R_2 - VOLTMETER MULTIPLIER
V - 0-100 VOLT WESTON COPPER OXIDE RECTIFIER VOLTMETER. 1000 OHMS PER VOLT
E - EXPLORING ELECTRODES MOUNTED IN TEST JIG

FIG. 1.—Testing Circuit.

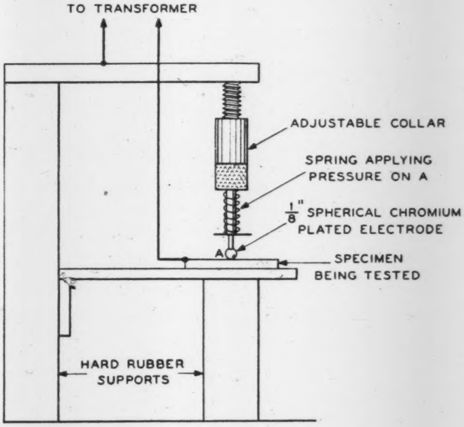


FIG. 2.—Test Jig.

APPARATUS

The testing circuit as shown in Fig. 1 is composed of a safety switch, a General Radio "Variac," a step-up transformer, a current-limiting resistor, a voltmeter, and the testing jig for holding the electrodes. A safety switch is provided to protect the operator from the high voltage and is so arranged that the circuit is opened and closed by the door to the test chamber. All of the high voltage part of the circuit is within the area guarded by the safety switch. A "Variac" having a capacity of 200 v-amp. has been found to be adequate and is mounted outside of the enclosed area for convenience in operation. Care must be taken to see that the connection between its output and the primary of the step-up transformer are protected from accidental contact with the high voltage. The step-up transformer is of the type used in radio plate power supplies and is capable of supplying about 50 ma. at 1500 v. The resistor R_1 is for the purpose of limiting the maximum current so as to prevent overloading of the transformers. The most practical type of voltmeter has been found to be the copper-oxide rectifier type having a resistance of 1000 ohms per volt. The scale of the meter is calibrated in root mean square alternating current volts. The multipliers for the voltmeter are simple radio type cartridge resistors sometimes referred to as grid leaks and are capable of carrying 1 ma. The structure of the test jig carrying the electrodes used by the authors is shown in Fig. 2.

A smooth, chromium-plated steel sphere, $\frac{1}{8}$ in. in diameter was used as the exploring electrode for most of the voltage breakdown tests. During a number of preliminary tests it was noted that too severe arcing, especially at high voltages, produced objectionable burning of the electrode, which had to be polished rather frequently in order to obtain fair duplicability. The entire set of test specimens was tested with a dead load on the electrode of 2.205 lb. (1000 g.). In addition, several anodic treatments were tested with no weight, and with 4.410 lb. (2000 g.) on the exploring electrode.

TABLE I.

Code	Anodizing Time, min.	Seal	Seal Time, min.	Current Density, amp. per sq.ft.	Base Alloy	Breakdown Voltage
10 per cent H_2SO_4, 95 F.						
1W	10	Water[a]	10	14.4	2S-1/2H; 2SO	210
2W	20		20			360
3W	30		30			490
4W	40		30			620
5W	50		30			750
6W	60		30			850
1U	10	None	....	14.4	2S-1/2H	200
3U	30					380
5U	50					580
1D	10	Chromate[b]	10	14.4	2S-1/2H	250
3D	30		30			500
5D	50		30			550
1S	10	Water[a]	10	14.4	2SO	120
3S	30		30			400
5S	50		30			700
1Y	30		30	5	2S-1/2H	170
2Y	30		30	10		320
3Y	30		30	20		780
4Y	30		30	25		1020
5Y	30		30	30		1170
6Y	30		30	35		1330
25 per cent H_2SO_4, 75 F.						
3W1	30	Water[a]	30	14.4	2S-1/2H	550
3U1	30	None	—			460
25 per cent H_2SO_4, 85 F.						
3A2	30	NiAc[c]	5	14.4	2S-1/2H	440
3W2	30	Water[a]	30			420

[a] Bath temperature 212 F.
[b] 5 per cent sodium dichromate solution at 212 F.
[c] Nickel acetate solution at 212 F.

METHOD OF TEST

With the specimen in place and the electrodes properly in contact with it, the dial of the "Variac" was turned slowly and smoothly, building up the voltage at such a rate that the needle of the voltmeter could follow the voltage change without lag. When breakdown was reached, the needle dropped to zero or some other low value. The maximum reading of the voltmeter was taken as the breakdown voltage. Twenty-five breakdown measurements were made on representative areas of each of the flat test panels and averaged to obtain the values listed in Table I.

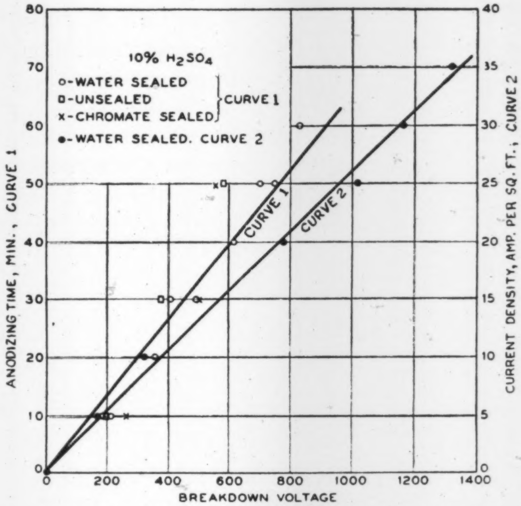


FIG. 3.- Relation of Anodizing Time and Current Density to Breakdown Voltage. 10 per cent H_2SO_4.

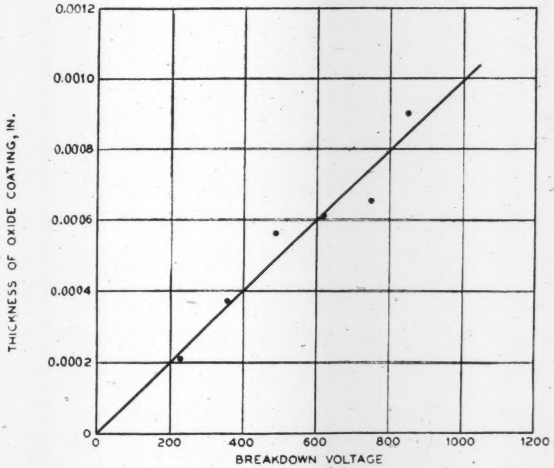


FIG. 4.—Relation of Thickness of Water-Sealed Oxide Coatings to Breakdown Voltage. 10 per cent H_2SO_4 at 95 F. Current density 14.4 amp. per sq. ft.

Test Specimens:

The specimens employed in this investigation were anodically oxidized by the commercially used Alumilite process,[2] and after the voltage breakdown tests were subjected to the abrasion tests described in a companion paper by H. G. Arlt.[3]

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[2] Protected by patents owned by the Aluminum Company of America.

Most of the above specimens consisted of $\frac{1}{16}$-in. panels which were polished with Tripoli and Learox No. 304 polishing compound and were degreased with acetone just prior to anodizing. In addition, a number of washers were tested which had been anodized for an earlier study. These washers were selected as representative of a product that had been anodized in racks and included the effects of racking, position in rack, and dyeing. The specimens used in the bending test were made of $\frac{1}{32}$-in. 2S sheet aluminum.

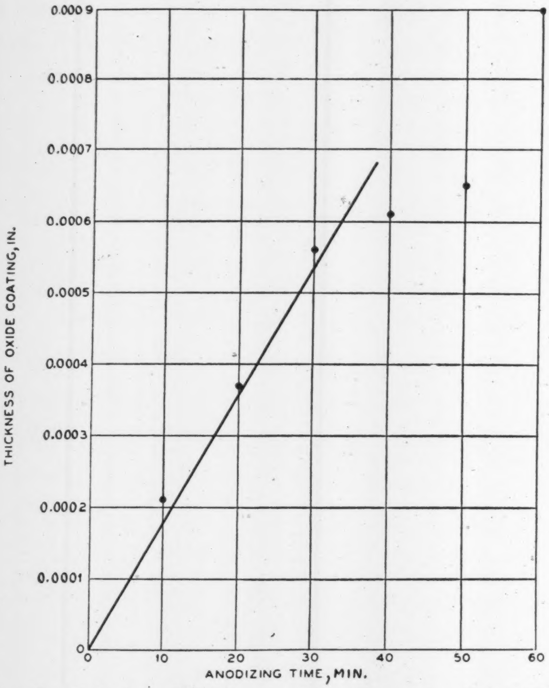

FIG. 5.—Relation of Thickness of Coating to Anodizing Time.
10 per cent H_2SO_4 at 95 F. Current density 14.4 amp. per sq. ft.

TEST RESULTS

Curve 1 of Fig. 3 represents the relationship between anodizing time and breakdown voltage, while curve 2 shows the breakdown voltage as a function of current density at a constant coating time of 30 min. In Fig. 4 the breakdown voltage is plotted against thickness of the water-sealed oxide coatings. Measurements of the oxide films with a calibrated high-powered microscope showed that the thickness is essentially a linear function of the anodizing time up to approximately 40 min. (see Fig. 5). These curves indicate that the breakdown voltage has a reasonably linear relation to anodizing time and consequently to thickness of coating up to a limiting value which for the conditions of these experiments seems to be about 40 min. Beyond this period the thickness is likely to be irregular and unreproducible. Although coatings of thicknesses less than 0.0002 in. have not been included in this study, it is believed that breakdown values in the lower ranges of thickness are apt to be erratic owing to contact difficulties that are likely to arise. The straight line of curve 2 likewise indicates that the rate of formation of the oxide coating is a direct function of the quantity of current (coulombs) passed through the cell, and bears in this respect a resemblance to the electrolytic deposition process for metals. The low value of the 50-min., chromate-sealed specimen lacks at present a satisfactory explanation. Check tests on duplicate samples have, however, confirmed the original result. The values obtained with the sample panels anodized in the high acid bath show that an increase in operating temperature is detrimental to the dielectric strength of the oxide film. Microscopic thickness measurements showed that the oxide film on 3W1 is 0.00058 in. against 0.00045 in. for the 3W2 specimen. This seems to indicate that a looser coating is produced at a higher temperature.

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[3] See p. 967.

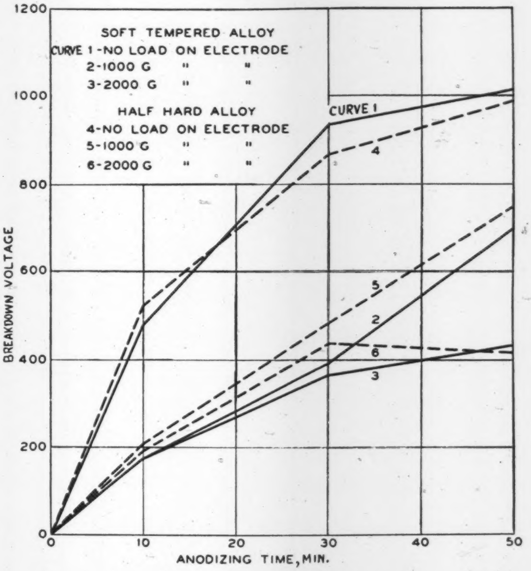

Fig. 6.—Results of Contact Pressure Study on 2S-$\frac{1}{2}$H and 2S-O Aluminum.

Effect of Pressure:

The contact pressure study on 2S-$\frac{1}{2}$H (half hard) and 2S-O (soft) aluminum alloys yielded the following results, which are also shown in Fig. 6:

	Panel	No load	1000 g.	2000 g.
Soft alloy........	1S	480 v.	180 v.	180 v.
	3S	940 v.	400 v.	370 v.
	5S	1020 v.	700 v.	430 v.
Half-hard alloy..	1W	530 v.	210 v.	200 v.
	3W	870 v.	490 v.	440 v.
	5W	1000 v.	750 v.	420 v.

These values indicate that for the thinner oxide films, as represented by 1S, 3S, 1W and 3W, doubling of the pressure on the exploring electrode has no significant effect; only in the range of thicker films (5S and 5W) is there a noticeable difference. As was to be expected, the values obtained with the unloaded electrode are of greater magnitude. Noteworthy is the fact that the hardness of the base metal has little influence on the breakdown of the anodic coating. The fairly constant slope of curves 2 and 5, Fig. 6, seem to indicate that a 1000-g. load on the exploring electrode is the optimum pressure to be employed, in view of the fact that thickness of oxide coating is proportional to anodizing time.

In order to obtain an idea of the pressure per square inch and the extent to which the panels were being dented with the variously loaded electrode, microscopic measurements of the contact impressions were made, yielding the following results:

Panel	Dead Load, g.	Actual Pressure, psi.	Diameter of Impression, in.	Approximate Depth of Impression, in.
1S....	1000	13 900	0.01423	0.0004
3S....		13 800	0.01428	0.0004
5S....		13 500	0.01443	0.0004
1W...		28 700	0.00950	0.0002
3W....		28 700	0.00950	0.0002
5W....		.....	....	......
1S....	2000	11 800	0.02180	0.0007
3S....		14 900	0.01942	0.0007
5S....		16 000	0.01870	0.0007
1W....		33 000	0.01300	0.0003
3W....		33 000	0.01305	0.0003
5W....		33 000	0.01300	0.0003

It is of interest to note that while the load on the electrode was doubled, the actual pressure per unit area remained approximately the same; the depth of the impression, on the other hand, doubled in value when the load was increased from 1000 g. to 2000 g. As has been seen, the effect on the breakdown resistance obtained by doubling the dead load on the electrode becomes noticeable only in the case of thicker coatings.

Effect of Bending:

In order to determine the effect of bending of the oxide film on its breakdown resistance, the following tests were made. Polished 4 by 6 by $\frac{1}{32}$-in. 2S-O (soft) aluminum alloy panels were treated in accordance with the methods outlined under 1W to 6W, inclusive. These panels were bent on a conical mandrel machine[4] and tested for voltage breakdown *in situ* using the small spherical exploring electrode. Check readings were made on the areas represented by the following mandrel sizes: $\frac{1}{8}$ (sharpest bend), $\frac{1}{2}$, $\frac{3}{4}$, $1\frac{1}{8}$ and $1\frac{1}{2}$ in. (minimum bend). Below are listed the results obtained from these tests:

Code	Breakdown Voltage				
	⅛-in. Mandrel[a]	½-in. Mandrel	¾-in. Mandrel	1⅛-in. Mandrel	1½-in. Mandrel
1W...	—	210	204	240	240
2W...	—	230	225	240	260
3W...	—	250	250	260	290
4W...	—	280	300	310	330
5W...	—	290	300	330	350
6W...	—	280	300	320	350

[a] The results obtained on the ⅛-in. mandrel were in every case interspersed with one or more zero readings, indicating that physical contact had been made with the underlying metal. The positive values obtained at this point were in general of the same order of magnitude as those obtained on the ½-in. mandrel.

The comparatively low and almost uniformly equal values of the voltage breakdown resistance over the entire thickness range, as compared to the progressively higher values for flat specimens, suggest the possibility that in effect what is being measured is the arcing voltage of the air gaps produced by cracking of the film. To partially verify this assumption, discharge voltages of two small air gaps were measured —approximately 0.0002 and 0.0004 in. in width. The following results were obtained:

For the 0.0002-in. gap:	For the 0.0004-in. gap:
260 v.	320 v.
250 v.	320 v.
260 v.	380 v.
300 v.	320 v.

Since these dimensions are of the order of magnitude of the thickness of oxide coatings, it is reasonable to assume that the air-gap hypothesis is partially correct. Values of the same magnitude were obtained by Loeb[5] for spark discharges in nitrogen.

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[4] H. G. Arlt, "Study of Conical Mandrel Test for Attached Lacquer Films," ASTM BULLETIN, No. 89, December, 1937, p. 5.

In addition to the breakdown measurements made with the small spherical electrode, several measurements were made with a tinfoil electrode 1 in. square, exerting a pressure of 4.22 psi. Intimate contact with the oxide coating was obtained by placing a sponge rubber cushion between the foil and the pressure-exerting weight. The treatment coded 3W was used for this experiment and gave the following results which confirm in a measure those obtained with the small steel-ball electrode, but it must be borne in mind that these values are minima and not average values and are not therefore directly comparable:

440 v.
440 v.
440 v.
500 v.
540 v.

480 v.
460 v.
460 v.
540 v.

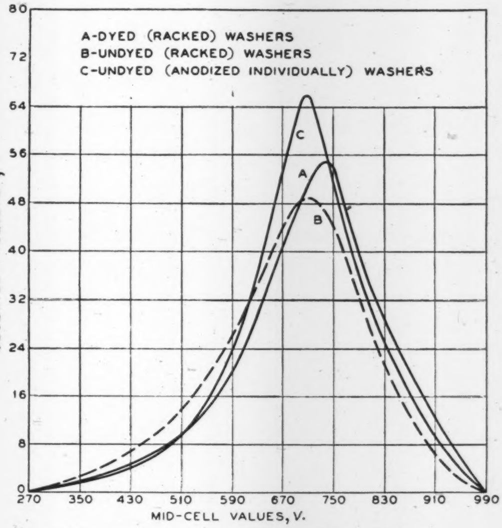

Fig. 7.—Distribution of Breakdown Voltages.

Statistical Analysis

The results described in the preceding pages are average values of a limited number of readings and are in view of the fair constancy, satisfactory within the limitations of the method. However in view of the large amount of data which could be obtained by this procedure, it is felt that a statistical analysis should afford a better picture of the distribution as well as of the range of the values than can be obtained by merely averaging the results. As an illustration of such a statistical analysis the following is presented.

The anodically oxidized washers previously mentioned were used in this part of the investigation. The method of analysis employed here is that described by Shewhart[6] which consists in grouping values of a certain range in so-called cells and plotting their mid-values against the frequencies of the observed values in each cell. From an inspection of the curve thus obtained, the distribution of values is readily ascertained. A narrow banded, steep peak is indicative of a good distribution of close-valued figures. Figure 7 shows a composite plot of three curves for comparison purposes. In addition to the graphical representations of the results, the arithmetic mean value for each individual sample and the standard deviation may be computed from the mid-cell values and the observed frequencies. Also, a calculation has been made of the percentage of the values lying within the range of the arithmetic mean plus and minus the standard deviation.

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[5] L. B Loeb, "The Problem of the Mechanism of Static Spark Discharge," *Reviews of Modern Physics*, Vol. 8, p. 278 (1936).

[6] W. A. Shewhart, "Economic Control of Quality of Manufactured Products," Chapters VII and VIII, D. van Nostrand Co., New York, N. Y. (1931).

The washers were in three groups of seventeen each, one group having been anodized and sealed in boiling water, a second group dyed with Alizarin Red S, while a third group was composed of individually anodized and water-sealed washers. Twenty breakdown measurements were made on each washer, using the ⅛-in. ball electrode and a load of about 1000 g.

Mid-Cell Value, v.	Deviation in Cells from σ*, X	Observed Frequency, Y	XY	X^2Y
310....	0	1	0	0
350....	1	4	4	4
390....	2	14	28	56
430....	3	5	15	45
470....	4	5	20	80
510....	5	18	90	450
550....	6	16	96	576
590....	7	35	245	1 715
630....	8	28	224	1 792
670....	9	41	369	3 320
710....	10	49	490	4 900
750....	11	41	451	4 960
790....	12	41	492	5 910
830....	13	20	260	3 380
870....	14	13	182	2 548
910....	15	7	105	1 124
950....	16	2	32	512
Σ		340	3103	31 372

* This means the unit deviation from the mid-cell value of the first cell, σ.

Sample Calculations:

The arithmetic mean, $\bar{X}$, is given by

$$\bar{X} = \sigma + M_1 u_1$$

where M = the unit per cell

$$= 40 \text{ v. and } {}_1u_1 = \frac{\Sigma XY}{\Sigma Y}$$

$$\text{then: } \bar{X} = 310 + \frac{3103}{340} \times 40 = 675 \text{ v.}$$

The standard deviation, σ, is given by

$$\sigma = M\sqrt{u_2}$$

$$\text{where } u_2 = {}_1u_2 - {}_1u_1^2 \text{ and } {}_1u_2 = \frac{\Sigma X^2 Y}{\Sigma Y}$$

$$\text{then: } \sigma = 40 \times \sqrt{\frac{31{,}372}{340} - \left(\frac{3103}{340}\right)^2}$$

The percentage of the values lying within the range $\bar{X} \pm \sigma$ (560 and 800 v.) has been found to be:

$$\frac{\text{Values between } \bar{X} \pm \sigma}{\Sigma Y} = \frac{248}{340} = 73.0 \text{ per cent}$$

By using the same method of calculation for all three groups the following results are obtained:

	Arithmetic Mean, $\bar{X}$, v.	Standard Deviation, σ, v.	Value Within Range, $\bar{X} \pm \sigma$, per cent
Undyed racked washers.....	675	120	73.0
Dyed racked washers........	718	119	76.1
Undyed individually anodized washers.............	698	108	69.8

The steepness and the similarity of shape of the curves in Fig. 7 are indicative of a satisfactory and fairly equal distribution of values around a mean value in all cases investigated. A further quantitative indication of the satisfactoriness of the distributions is evidenced by the values of the arithmetic mean, the standard deviation, and percentage of values lying between $\bar{X} \pm \sigma$ computed from the group data for each individual condition.

CONCLUSION

The authors have endeavored to show that the thickness of coatings on anodically oxidized aluminum articles can be satisfactorily evaluated by means of a rapid, practically non-destructive method. While it cannot be regarded as a primary means of measuring the thickness it should be looked upon mainly as a "checking" of the finish on anodized parts without too much labor and loss of time. It furthermore should be noted that only one alloy has been investigated and that the oxide coatings were produced on polished surfaces only.

Undoubtedly different alloys and surface preparations are likely to have a marked influence on the breakdown resistance of the oxide coatings. A comprehensive study of the subject should, therefore, include all these variables. Although, for obvious reasons, the present investigation had to be limited in its scope, it indicates the applicability of this method of evaluating thicknesses of oxide coatings to all conditions.

As a result of this investigation the authors now employ voltage breakdown in the laboratory as a routine means of checking the thickness of aluminum oxide coatings with satisfactory results. A number of measurements on a sample can be taken and averaged within a few seconds as compared with the much longer time required by other methods. *For greater precision a calibration is made for the particular conditions under which the samples have been finished,* but for general work a rough factor of 93 v. per 0.0001 in. has been used. It has been found that the thicknesses obtained by this method are generally well within 10 per cent of the values determined with the microscope. Care must be taken to avoid measurements too near an edge or at some point where the coating has been subjected to bending or to base conclusions upon too small a number of measurements. In most cases the measurements can be made without destroying the sample by proper selection of the point at which the test is applied.

TESTS OF 106 COMMERCIAL CEMENTS FOR SULFATE RESISTANCE[1]

By Dalton G. Miller[2] and Philip W. Manson[3]

Synopsis

Results of 5-yr. sulfate resistance tests of 5724 concrete cylinders, 2 by 4 in., made of 106 commercial cements from 74 mills are recorded in this report. Some of the cylinders were continuously stored in the laboratory in 1 per cent solutions of magnesium sulfate (MgS_4), some in 1 per cent solutions of sodium sulfate (Na_2SO_4), and some in the water of Medicine Lake, S. Dak. During the 5-yr. period, the water of this lake averaged 12 per cent total salts, of which two-thirds were magnesium sulfate and one-fourth sodium sulfate. Resistance of the specimens stored in the laboratory solutions was determined by length changes while resistance of the Medicine Lake cylinders was determined by strength ratios calculated from compression tests at 1 and 5 yr. of the Medicine Lake cylinders and cylinders stored in tap water in the laboratory.

The many tests and chemical analyses indicate close correlation between sulfate resistance of the cements and the percentage of the calculated compound tricalcium-aluminate.

This progress report covers results of 5-yr. tests of 5724 concrete cylinders made of 106 commercial cements from 74 mills. The experiments, while originally planned to aid in the general improvement of farm drain tile, have a wide application to all other types of construction in which portland cement concretes and mortars, in service, must resist the action of soils or waters rich in sulfates.

Test Specimens.—The specimens on which the work is based were cylinders 2 in. in diameter and 4 in. long. The aggregate had a fineness modulus of 4.67 and weighed 124 lb. per cu. ft., dry-rodded. All cylinders were of a 1:3 mix with a relative consistency of 1.00 and a water-cement ratio of 0.62 to 0.66, corresponding to about 4.75 gal. per bag of cement. All cylinders exposed to the laboratory solutions were immersed in the solutions at 21 days, following curing in the moist closet for 24 hr. and water for 20 days. The Medicine Lake cylinders were similarly cured plus 35 days in air in the laboratory.

Exposure.—Five cylinders of all cements were continuously stored in the laboratory in 1 per cent solutions of magnesium sulfate ($MgSO_4$), 5 cylinders in 1 per cent solutions of sodium sulfate (Na_2SO_4), and 20 cylinders 4 ft. deep in

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[1] *Paper No. 1821,* Scientific Journal Series, Minnesota Agricultural Experiment Station.

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TABLE I.—RESULTS OF TESTS MADE IN CONNECTION WITH THE DETERMINATION OF SULFATE RESISTANCE OF THE 106 CEMENTS OF TABLE II.

The 106 cements in the 11 groups are arranged in descending order of resistance based on averages for the three exposure conditions.

Figures in the months columns of the expansion results indicate the time required to increase in length 0.01 in

Figures in parentheses indicate percentage of normal strength of the Medicine Lake cylinders based on parallel tests of cylinders from the same batches stored in tap water.

Test results of cylinders are in all cases averages for 5 cylinders made on different days.

		Strength Tests									Expansion Tests			
		Tension Tests of Standard Briquets, psi.			Compression Tests of 2 by 4-in. Cylinders, psi.						Increase in Length of 2 by 4 in. Cylinders Stored in Laboratory Solutions			
		Moist Closet			Tank Specimens				Medicine Lake Specimens		1 per cent $MgSO_4$		1 per cent Na_2SO_4	
Series	Cement	3 days	7 days	28 days	7 days	28 days	1 yr.	5 yr.	1 yr.	5 yr.	inch	months	inch	months
GROUP 1														
1584....	611...	260	335	390	4310	6160	9600	8710	8010 (83)	7410 (85)	0.000		0.000	
1599....	631...	310	370	445	4680	5120	9040	8750	8050 (89)	8030 (92)	0.001		0.001	
1551....	531...	185	255	355	2810	5230	8130	8940	7080 (87)	7820 (88)	0.002		0.000	
1511....	405...	275	340	415	4030	5820	7120	8490	7060 (99)	6720 (79)	0.000		0.001	
1568....	591...	240	295	345	3910	4180	7010	7660	6440 (92)	6110 (80)	0.001		0.001	
1571....	576...	200	...	315	3220	5080	6830	8370	7690 (113)	6230 (74)	0.001		0.001	
1518....	426...	240	295	380	3890	5370	8750	9620	7820 (89)	6620 (69)	0.001		0.001	
1596....	628...	300	360	410	4410	5730	8130	9440	8120 (100)	7860 (83)	0.002		0.001	
1576 ...	582...	225	295	290	3770	4830	7650	7980	6720 (88)	6600 (83)	0.001		0.002	
1580....	596...	300	375	445	5130	5760	8110	8730	8510 (105)	5960 (68)	0.002		0.000	
Average		255	325	380	4020	5330	8040	8670	7550 (95)	6940 (80)	0.001		0.001	
GROUP 2														
1505....	387...	315	410	445	5230	5980	7380	8650	6470 (88)	7020 (81)	0.002		0.002	
1593....	621...	130	...	460	2710	4880	8450	7640	8060 (95)	4810 (63)	0.001		0.001	
1506....	390...	280	325	380	4400	5200	7480	7690	6100 (82)	6620 (86)	0.003		0.002	
1543....	507...	275	345	465	4130	5940	8670	9180	7610 (88)	7040 (77)	0.002		0.002	
1583....	605...	275	305	410	4300	5420	8640	9150	8040 (93)	6920 (76)	0.002		0.002	
1563....	561...	320	430	470	5010	6520	7850	8380	7340 (94)	5070 (61)	0.001		0.002	
1507....	393...	355	390	445	5720	6400	7670	7850	5910 (77)	4900 (63)	0.003		0.002	
1537....	489...	255	300	370	3880	5630	7780	9000	7290 (94)	5930 (66)	0.003		0.003	
1554....	543...	250	370	400	4800	6040	8600	9310	8060 (94)	6240 (67)	0.003		0.003	
1547....	516...	295	355	445	4920	6680	7480	9050	7570 (101)	6360 (70)	0.003		0.004	
Average		275	360	430	4510	5870	8000	8590	7250 (91)	6090 (71)	0.002		0.002	
GROUP 3														
1503....	381...	220	315	385	3690	5970	6840	7000	6550 (96)	6040 (86)	0.003		0.005	
1579....	608...	235	305	410	4780	6160	8960	8770	7350 (82)	5460 (62)	0.003		0.003	
1546....	513...	265	375	385	4150	5080	7800	8190	7190 (92)	5690 (70)	0.003		0.004	
1523....	441...	275	360	430	4000	5520	7700	9390	5990 (78)	2810 (30)	0.001		0.002	
1552....	534...	375	445	485	5390	6670	9120	9160	7320 (80)	4920 (54)	0.007		0.002	
1508....	396...	350	390	460	5790	6130	5810	8980	6890 (119)	3480 (39)	0.003		0.004	
1548....	519...	425	475	465	6060	6680	8140	9410	6780 (83)	4550 (48)	0.004		0.004	
1553....	540...	220	310	345	4870	5950	8360	8740	7070 (85)	2350 (27)	0.004		0.002	
1550....	528...	245	355	425	4400	5930	7940	9180	7230 (91)	4720 (52)	0.006		0.003	
1502....	378...	285	345	430	4800	6020	6820	9120	5480 (80)	3210 (35)	0.003		0.004	
Average		290	370	420	4790	6010	7750	8790	6790 (89)	4320 (50)	0.004		0.003	
GROUP 4														
1520....	432...	260	315	330	4120	5650	7890	8550	6290 (80)	3590 (42)	0.004		0.005	
1536....	486...	235	325	370	3780	5650	6800	8230	6640 (98)	4960 (60)	0.007		0.006	
1566....	570...	420	520	500	6920	7290	8190	8820	7120 (87)	3600 (41)		65	0.005	
1524....	444...	430	420	445	5140	5890	7600	6520	5990 (79)	1570 (24)	0.004		0.004	
1521....	435...	240	325	365	3860	5220	6920	8740	5830 (84)	3070 (35)	0.005		0.007	
1549....	525...	305	380	410	5290	5910	8420	9580	7900 (94)	4720 (49)		65		55
1514....	414...	265	375	395	5320	6780	7560	7930	6410 (85)	1290 (16)	0.004		0.004	
1587....	614...	240	320	440	4340	6010	8310	8080	5890 (71)	660 (8)	0.006		0.003	
1532....	474...	325	380	415	5050	5690	7900	7840	7210 (91)	3410 (44)		55		57
1531....	471...	280	335	395	4420	5080	8160	8000	6090 (75)	4220 (53)		53		56
Average		300	370	405	4820	5920	7780	8230	6540 (84)	3110 (37)				

(*Continued on p. 990*)

TABLE I.—*Continued.*

Series	Cement	Strength Tests									Expansion Tests			
		Tension Tests of Standard Briquets, psi.			Compression Tests of 2 by 4-in. Cylinders, psi.						Increase in Length of 2 by 4-in. Cylinders Stored in Laboratory Solutions			
		Moist Closet			Tank Specimens				Medicine Lake Specimens		1 per cent $MgSO_4$		1 per cent Na_2SO_4	
		3 days	7 days	28 days	7 days	28 days	1 yr.	5 yr.	1 yr.	5 yr.	inch	months	inch	months
GROUP 5														
1567....	590...	225	310	375	3750	4880	7260	7160	5890 (81)	2350 (33)		53	0.005	
1564....	564...	215	320	390	4120	5760	9240	8690	6570 (71)	2790 (32)		57		55
1541....	498...	425	440	460	5720	6530	8440	8930	7390 (88)	2290 (26)	0.006			53
1594....	624...	250	245	385	3980	5870	7030	8310	6300 (90)	2430 (29)		51	0.005	
1512....	408...	310	375	455	4800	6590	7200	7170	6040 (84)	2700 (38)		46		60
1504....	384...	255	350	405	4440	6300	7260	8680	6120 (84)	2640 (30)		60		43
1558....	546...	275	345	395	5030	6120	8890	8180	5920 (67)	2180 (27)		55		49
1545....	510...	245	350	415	4200	5230	8110	7970	7160 (88)	1960 (25)		54		51
1555....	462...	320	370	425	4770	7100	9310	10030	7880 (85)	1710 (17)		51		55
1562....	558...	260	350	405	4160	6000	8040	9100	6710 (83)	1570 (17)		54		51
Average		275	345	410	4500	6040	8080	8420	6600 (82)	2260 (27)				
GROUP 6														
1570....	573...	280	370	385	4270	6190	8000	9420	6540 (82)	1420 (15)		50		54
1527....	453...	265	360	400	4920	5920	6570	8450	5500 (84)	1320 (16)		53		46
1509....	399...	295	350	390	4650	5840	6630	8130	8290 (125)	1020 (13)		48		46
1565....	567...	335	380	410	5240	6430	8370	8200	6850 (82)	550 (7)		45		48
1522....	438...	255	345	405	4260	5520	6400	7860	5750 (90)	0		46		53
1561....	555...	325	405	425	5960	6980	8410	8120	6270 (75)	0		54		39
1581....	599...	290	340	405	5450	6120	6610	6620	5630 (85)	0		41		48
1559....	549...	370	410	375	5360	6940	7620	7870	5670 (74)	0		44		47
1510....	402...	240	370	405	4850	6180	7430	9030	5270 (71)	0		49		42
1557....	465...	290	355	375	4450	5860	8740	9500	6590 (75)	0		48		41
Average		295	370	400	4940	6200	7480	8320	6240 (84)	490 5		48		46
GROUP 7														
1539....	501...	290	375	400	5610	6890	7880	8860	6700 (85)	0		47		37
1538....	492...	315	365	440	4930	6480	7880	8180	5900 (75)	0		47		44
1591....	619...	280	345	400	3760	4720	7460	8120	6030 (81)	0		43		41
1529....	459...	320	400	405	5430	6350	7210	8590	5970 (83)	0		41		43
1517....	423...	255	355	420	4140	5320	6410	8610	5010 (78)	0		38		40
1540....	495...	260	315	355	4500	5950	8190	8910	5720 (70)	0		42		27
1533....	477...	260	355	385	5280	6260	8050	8910	6150 (76)	0		37		27
1597....	629...	385	490	505	5930	8730	9100	8090	6380 (70)	0		34		37
1526....	450...	370	420	440	4600	4910	6160	7140	4200 (68)	0		35		29
1556....	552...	435	505	445	5490	7320	7810	8010	5060 (65)	0		37		26
Average		315	395	420	4965	6290	7620	8340	5710 (75)	0		40		35
GROUP 8														
1516....	420...	380	410	430	5770	6410	7220	7700	4710 (65)	0		35		34
1626....	663...	230	360	415	4420	5780	5960	7140	5420 (92)	0		31		24
1525....	447...	280	360	385	5060	6020	7370	8690	5190 (70)	0		30		29
1632....	669...	320	410	435	5370	6200	7500	7460	4630 (62)	0		33		27
1588....	615...	250	305	405	4270	5580	7690	7510	4460 (58)	0		30		41
1592....	620...	310	365	435	4980	6270	7340	8170	5540 (75)	0		28		27
1590....	618...	340	400	440	5660	6560	8430	7680	6280 (74)	0		25		31
1595....	627...	270	310	425	4190	5860	8130	8240	6340 (78)	0		27		18
1528....	456...	340	380	395	5820	6270	7050	7940	4310 (61)	0		30		26
1542....	504...	265	340	420	4290	5330	8030	9970	5150 (64)	0		29		17
Average		300	365	420	4980	6030	7470	8050	5200 (70)	0		30		27

(*Continued on p. 991.*)

TABLE I.—*Continued.*

Series	Cement	Strength Tests									Expansion Tests			
		Tension Tests of Standard Briquets, psi.			Compression Tests of 2 by 4-in. Cylinders, psi.						Increase in Length of 2 by 4-in. Cylinders Stored in Laboratory Solutions			
		Moist Closet			Tank Specimens				Medicine Lake Specimens		1 per cent $MgSO_4$		1 per cent Na_2SO_4	
		3 days	7 days	28 days	7 days	28 days	1 yr.	5 yr.	1 yr.	5 yr.	inch	months	inch	months
GROUP 9														
1618....	645...	320	345	390	5330	6470	5670	7300	4740 (84)	0		26		26
1609....	641...	330	430	455	4770	6690	6670	7770	4910 (74)	0		35		20
1623....	656...	285	350	385	5050	5550	6960	8030	5040 (72)	0		32		20
1582....	602...	270	330	405	5220	6090	6680	8510	5510 (82)	0		23		24
1621....	650...	235	315	355	4770	6100	6780	7860	4890 (72)	0		27		21
1573....	594...	305	350	470	3600	4520	7580	7230	2450 (32)	0		29		28
1617....	644...	250	335	370	4150	6480	6280	7490	4150 (66)	0		24		22
1500....	372...	260	310	410	4060	5340	7680	8440	4760 (62)	0		32		17
1575....	579...	255	325	420	5070	6370	8140	8760	5820 (71)	0		21		21
1560 ...	537...	235	345	355	4180	5880	7430	7660	4700 (63)	0		31		16
Average		275	345	400	4620	5950	6990	7910	4700 (68)	0		28		22
GROUP 10														
1641....	685...	335	375	355	4500	5420	5990	7570	5810 (97)	0		25		17
1574....	595...	255	305	350	3970	3990	5600	5530	3320 (59)	0		30		21
1513....	411...	250	340	380	5000	6010	6540	6800	4770 (73)	0		27		16
1501....	375...	290	375	410	5640	5960	7290	8070	4060 (56)	0		29		14
1631....	668...	260	335	420	4510	5560	6070	7800	4420 (73)	0		21		14
1534....	480...	330	375	425	5490	6620	8650	7460	5360 (62)	0		23		14
1544....	522...	305	390	405	5350	5710	7440	8570	4860 (65)	0		24		12
1515....	417...	275	345	350	5700	5330	7100	7140	3700 (52)	0		23		14
1600....	632...	300	390	420	4880	5560	7970	7590	3710 (47)	0		23		13
1519....	429...	350	370	380	4530	5820	7830	8770	2660 (34)	0		19		13
Average		295	360	390	4960	5600	7050	7530	4270 (62)	0		24		19
GROUP 11														
1572....	593...	275	360	425	4700	5670	7350	7900	3080 (42)	0		16		12
1530....	468...	285	325	360	4260	5020	6860	8040	2990 (44)	0		16		10
1615....	680...	295	380	420	4950	6710	7090	7140	4860 (69)	0		11		10
1598....	630...	240	305	355	3870	5180	7540	7760	3430 (45)	0		15		10
1625....	662...	265	380	410	4380	6370	6700	7990	2700 (40)	0		12		11
1535....	483...	255	295	395	4070	5210	7040	7750	1990 (28)	0		16		10
Average		270	340	395	4370	5690	7100	7760	3180 (45)	0		14		11

Medicine Lake, 18 miles northwest of Watertown, S. Dak. This lake averaged, during the 5-yr. period, 12 per cent total salts of which two-thirds were magnesium sulfate and one-fourth sodium sulfate, the remainder being a mixture of various other salts.

Method of Measuring Sulfate Resistance.—Resistance of the cylinders in the laboratory solutions was determined by length changes measured to 0.0001 in. by an Ames dial apparatus. Any cylinder that increased in length by as much as 0.01 in. was assumed to have completely failed and the corresponding age in months recorded. Resistance of the Medicine Lake cylinders was determined by strength ratios at 1 and 5 yr. based on strengths at 1 and 5 yr. of cylinders stored in tap water in the laboratory. All length changes and compression tests, as recorded, are averages for 5 cylinders made on different days. Equal weight was given results of the three tests, namely, (1) length increases of the cylinders in the 1 per

TABLE II.—OXIDE ANALYSES, CALCULATED COMPOUNDS, AND FINENESS DETERMINATIONS OF 106 COMMERCIAL CEMENTS TESTED FOR SULFATE RESISTANCE.

The 106 cements in the 11 groups are arranged in descending order of resistance as recorded in Table I.

Oxide analyses and fineness determinations were made during 1935–37 for the Bureau of Agricultural Engineering of the U. S. Department of Agriculture by the National Bureau of Standards.

T indicates a trace.

			Oxide Analyses, per cent														Ratio		Percentage Retained on			Calculated Compounds, per cent					
Series	Cement	Loss on Ignition	SiO_2	Fe_2O_3	FeO	TiO_2	P_2O_5	Mn_2O_3	Al_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	Insoluble Residue	Free Lime CaO	Fe_2O_3 to Al_2O_3	Al_2O_3 to Fe_2O_3	Sieve No. 200	Sieve No. 325	Surface Area, sq. cm. per g.	C_3S	C_2S	C_3A	C_4AF	$CaSO_4$	Sum of C_3A and C_4AF
GROUP 1																											
1584...	611...	1.2	22.5	3.7	0.02	0.25	0.17	0.06	4.4	64.1	1.4	0.28	0.51	1.7	0.06	0.4	0.84	1.19	4.9	13.6	1490	48.4	28.1	5.4	11.3	2.9	16.7
1599...	631...	1.4	20.4	4.8	0.00	0.63	0.10	0.08	4.3	63.8	1.9	0.36	0.20	1.9	0.10	0.3	1.12	0.90	5.4	12.1	1450	62.2	11.7	3.3	14.6	3.2	17.9
1551...	531...	1.2	23.2	4.5	T	0.19	0.12	0.13	4.2	62.1	2.4	0.16	0.26	1.7	0.12	0.1	1.07	0.93	12.5	23.6	1450	36.4	39.1	3.5	13.7	2.9	17.2
1511...	405...	2.2	22.3	2.9	T	0.24	0.12	0.10	3.8	63.7	2.3	0.15	0.51	1.7	0.20	1.1	0.76	1.31	4.5	12.5	1630	50.7	25.8	5.1	8.8	2.9	13.9
1568...	591...	1.4	19.9	5.2	0.02	0.25	0.11	0.06	4.8	63.8	1.8	1.20	0.02	1.8	0.12	0.9	1.08	0.92	8.5	20.3	1470	59.9	11.9	3.9	15.8	3.1	19.9
1571...	576...	1.3	23.6	3.9	T	0.20	0.06	0.01	3.9	63.0	2.2	0.20	0.51	1.4	0.20	0.8	1.00	1.00	10.5	21.6	1380	37.9	39.1	3.7	11.9	2.4	15.6
1518...	426...	1.1	22.8	3.5	0.00	0.23	0.19	0.47	3.8	63.0	2.6	0.19	0.44	1.8	0.15	0.4	0.92	1.09	2.6	8.0	1750	45.7	31.0	4.1	10.6	3.1	14.7
1596...	628...	1.1	21.8	5.2	0.02	0.22	0.29	0.05	2.8	65.3	1.2	0.67	0.13	1.7	0.14	1.3	1.86	0.54	3.5	10.6	1660	62.5	15.3	1.4[a]	13.4	2.9	....
1576...	582...	1.1	20.2	4.6	0.30	0.25	0.14	0.03	4.9	64.1	2.2	0.50	0.11	1.6	0.10	1.0	0.94	1.07	9.5	30.0	1160	59.2	13.4	5.2	14.0	2.7	19.2
1580...	596...	1.9	22.8	2.0	0.01	0.22	0.07	0.15	3.8	65.9	1.6	0.19	0.23	1.6	0.28	0.9	0.53	1.90	5.0	12.9	1670	58.1	21.6	6.7	6.1	2.7	12.8
Average.....		1.4	22.0	4.0	0.04	0.27	0.14	0.11	4.1	63.9	2.0	0.39	0.29	1.7	0.15	0.7	1.01	1.09	6.7	16.5	1510	52.1	23.7	4.5	12.0	2.9	16.5
GROUP 2																											
1505...	387...	1.1	22.9	2.8	T	0.26	0.03	0.04	3.9	65.5	1.1	0.19	0.41	1.9	0.05	0.8	0.72	1.39	4.3	10.0	1820	53.5	25.4	5.6	8.5	3.2	14.1
1593...	621...	3.5	22.0	5.4	0.09	0.22	0.13	0.03	4.4	58.2	3.3	0.44	0.67	1.8	0.06	0.1	1.23	0.82	4.4	11.6	1690	26.8	42.9	2.5	16.4	3.1	18.9
1506...	390...	1.2	22.4	3.0	T	0.21	0.07	0.09	3.9	63.2	3.6	0.25	0.50	1.9	0.17	1.4	0.77	1.30	2.0	14.0	1700	45.2	30.2	5.3	9.1	3.2	14.4
1543...	507...	1.4	22.2	4.0	T	0.15	0.08	0.06	3.4	62.3	4.1	0.11	0.33	1.8	0.08	0.6	1.18	0.85	2.7	7.4	1750	48.6	27.1	2.2	12.2	3.1	14.4
1583...	605...	1.0	21.6	5.0	0.00	0.24	0.10	0.07	4.3	62.1	3.3	0.25	0.73	1.8	0.12	0.1	1.16	0.86	3.3	8.4	1660	46.9	26.6	2.9	15.2	3.1	18.1
1563...	561...	4.1	20.8	2.9	0.25	0.34	0.37	0.08	4.2	62.2	2.2	0.44	0.18	1.9	0.36	1.0	0.69	1.45	3.4	6.7	2220	53.1	19.6	6.2	8.8	3.2	15.0
1507...	393...	1.4	21.7	3.1	T	0.25	0.05	0.10	3.6	63.0	3.9	0.31	0.39	2.3	0.17	0.7	0.86	1.16	2.2	5.8	2250	53.3	22.1	4.3	9.4	3.9	13.7
1537...	489...	0.9	21.5	4.6	T	0.28	0.04	0.07	4.8	63.4	1.9	0.20	0.69	1.6	0.13	0.2	0.96	1.04	3.6	10.9	1610	50.3	23.7	4.9	14.0	2.7	18.9
1554...	543...	1.8	21.1	4.2	T	0.24	0.13	0.07	4.2	63.9	2.3	0.15	0.28	1.7	0.12	1.3	1.00	1.00	7.0	14.1	1690	55.3	18.9	4.0	12.8	2.9	16.8
1547...	516...	0.9	22.3	2.6	T	0.25	0.10	0.08	5.3	64.1	2.1	0.18	0.51	1.8	0.19	0.5	0.49	2.04	7.5	16.8	1520	44.7	30.3	9.7	7.9	3.1	17.6
Average.....		1.7	21.9	3.8	0.03	0.24	0.10	0.06	4.2	62.8	2.8	0.25	0.47	1.9	0.15	0.7	0.91	1.19	4.0	10.6	1790	47.8	26.7	4.8	11.4	3.2	16.2
GROUP 3																											
1503...	381...	0.6	23.1	3.3	T	0.24	0.02	0.06	3.7	63.1	4.1	0.14	0.48	1.3	0.12	0.3	0.89	1.12	4.4	13.1	1650	46.6	31.2	4.2	10.0	2.2	14.2
1579...	608...	1.2	22.9	3.7	0.01	0.23	0.07	0.07	4.5	63.8	1.3	0.26	0.68	1.4	0.06	0.3	0.82	1.22	4.2	12.4	1560	44.8	31.9	5.6	11.3	2.4	16.9
1546...	513...	1.7	20.0	6.0	0.05	0.22	0.20	0.04	4.1	59.9	5.4	0.53	0.29	1.7	0.16	0.4	1.46	0.68	6.5	12.3	1910	49.2	20.3	0.7	18.2	2.9	18.9
1523...	441...	1.7	21.1	3.0	0.00	0.28	0.14	0.11	4.6	64.3	2.2	0.39	0.40	1.8	0.18	0.7	0.65	1.53	8.4	16.9	1480	58.0	16.8	7.1	9.1	3.1	16.2
1552...	534...	1.5	21.7	2.0	0.04	0.19	0.09	0.04	3.8	66.2	2.4	0.00	0.12	2.1	0.10	0.5	0.53	1.90	3.7	3.9	2560	67.9	11.0	6.7	6.1	3.6	12.8
1508...	396...	1.0	21.3	4.0	0.07	0.30	0.03	0.06	4.6	62.6	3.2	0.36	0.75	2.0	0.07	0.1	0.87	1.15	2.9	8.9	1880	50.1	23.4	5.4	12.2	3.4	17.6
1548...	519...	1.3	19.8	2.9	T	0.24	0.09	0.10	4.7	65.0	3.2	0.13	0.23	2.5	0.17	0.8	0.62	1.62	2.7	5.1	2530	67.9	5.6	7.5	8.8	4.3	16.3
1553...	540...	2.4	21.7	2.3	T	0.22	0.18	0.07	4.2	65.0	1.9	0.12	0.35	1.6	0.11	1.5	0.55	1.83	8.1	16.3	1660	57.3	19.1	7.2	7.0	2.7	14.2
1550...	528...	2.6	22.0	2.6	0.03	0.19	0.07	0.05	4.2	63.6	2.9	0.12	0.16	1.5	0.23	1.2	0.62	1.62	6.1	12.6	1750	50.4	25.1	6.7	7.9	2.6	14.6
1502...	378...	2.3	20.5	2.2	0.03	0.25	0.03	0.05	4.7	62.6	4.8	0.40	0.72	1.6	0.35	1.8	0.47	2.14	1.4	13.7	1500	52.2	19.5	8.7	6.7	2.7	15.4
Average.....		1.6	21.4	3.2	0.02	0.24	0.09	0.07	4.3	63.6	3.1	0.25	0.42	1.8	0.16	0.8	0.75	1.48	4.8	11.5	1850	54.4	20.4	6.0	9.7	3.0	15.7

Group 4																											
1520...	432...	2.4	20.4	3.0	T	0.25	0.21	0.30	4.7	63.6	2.8	0.41	0.51	2.0	0.15	2.2	0.64	1.57	5.5	14.7	1610	53.2	18.5	7.4	9.1	3.4	16.5
1536...	486...	1.5	21.4	3.0	T	0.31	0.05	0.05	5.3	64.1	2.1	0.11	0.63	1.5	0.37	0.4	0.57	1.77	4.3	11.9	1540	52.3	22.0	9.0	9.1	2.6	18.1
1566...	570...	1.9	19.0	3.6	T	0.28	0.18	0.28	6.2	65.0	1.0	0.29	0.32	2.4	0.08	0.5	0.58	1.72	0.3	0.6	2280	64.4	6.0	10.3	10.9	4.1	21.2
1524...	444...	1.7	20.4	2.9	T	0.30	0.14	0.11	4.6	65.4	2.2	0.48	0.27	1.7	0.14	0.9	0.63	1.59	0.7	2.2	2360	67.5	7.7	7.3	8.8	2.9	16.1
1521...	435...	1.8	20.9	2.2	T	0.64	0.09	0.04	5.0	64.5	3.0	0.32	0.23	1.7	0.19	2.2	0.44	2.28	3.6	17.9	1590	53.0	20.0	9.5	6.7	2.9	16.2
1549...	525...	1.3	20.9	2.7	0.16	0.27	0.29	0.14	5.4	63.3	3.0	0.06	0.52	1.9	0.42	0.9	0.50	2.00	3.1	-6.3	1930	49.4	22.7	9.7	8.2	3.2	17.9
1514...	414...	1.5	21.8	2.6	T	0.29	0.10	0.07	5.3	65.0	1.2	0.11	0.47	1.8	0.08	1.3	0.49	2.04	9.7	19.4	1590	49.0	25.6	9.6	7.9	3.1	17.5
1587...	614...	3.0	23.6	0.4	0.00	0.22	0.00	T	3.6	66.3	1.0	0.00	0.20	2.0	0.14	3.0	[b]	[b]	1.8	6.1	2140	47.6	31.8	8.9	1.2	3.4	10.1
1532...	474...	2.2	20.8	3.1	T	0.29	0.19	0.15	5.6	63.7	1.4	0.04	0.41	2.0	0.14	0.9	0.55	1.81	3.4	8.8	1830	49.6	22.3	9.6	9.4	3.4	19.0
1531...	471...	1.1	20.9	5.0	T	0.25	0.16	0.05	5.0	61.4	2.8	0.07	1.22	1.8	0.09	0.5	1.00	1.00	5.8	14.0	1700	43.1	27.5	4.8	15.2	3.1	20.0
Average.....		1.8	21.0	2.9	0.02	0.31	0.14	0.12	4.6	64.2	2.1	0.19	0.48	1.9	0.17	1.3	0.60	1.75	3.8	10.2	1850	52.9	20.4	8.6	8.7	3.2	17.3
Group 5																											
1567...	590...	2.1	21.4	2.6	0.02	0.26	0.12	0.06	5.3	63.9	1.7	1.20	0.02	1.7	0.26	0.7	0.49	2.04	6.0	16.9	1430	50.2	23.5	9.7	7.9	2.9	17.6
1564...	564...	1.3	22.0	3.5	T	0.22	0.03	0.55	4.5	64.5	1.7	0.23	0.18	1.6	0.12	0.2	0.78	1.29	4.4	12.7	1640	54.6	22.0	6.0	10.6	2.7	16.6
1511...	498...	1.6	20.2	3.2	T	0.24	0.20	0.25	5.5	63.2	2.2	0.23	0.62	2.5	0.10	0.8	0.58	1.72	1.0	1.3	2600	51.7	19.0	9.1	9.7	4.3	18.8
1594...	624...	1.7	21.2	2.7	0.01	0.33	0.01	0.04	4.9	62.1	4.2	0.10	0.77	2.0	0.26	1.1	0.55	1.82	8.5	17.4	1750	44.6	27.2	8.4	8.2	3.4	16.6
1512...	408...	1.9	20.0	5.5	0.00	0.30	T	0.69	5.8	61.3	2.4	0.30	0.09	1.7	0.13	0.3	0.95	1.05	5.8	14.4	1880	44.6	23.8	6.0	16.7	2.9	22.7
1504...	384...	1.7	21.6	2.4	T	0.23	0.02	0.09	5.2	62.9	3.3	0.16	0.59	1.8	0.15	0.7	0.46	2.17	6.9	17.5	1630	45.4	27.8	9.7	7.3	3.1	17.0
1558...	546...	1.3	20.5	3.6	0.05	0.27	0.08	0.05	5.7	63.5	2.5	0.18	0.63	1.8	0.15	0.9	0.63	1.58	3.2	9.2	1640	50.3	20.9	9.0	10.9	3.1	19.9
1545...	510...	1.1	22.1	2.3	T	0.19	0.24	0.04	4.7	62.4	4.6	0.49	0.28	1.8	0.26	0.6	0.49	2.04	5.5	13.6	1750	43.4	30.7	8.6	7.0	3.1	15.6
1555...	462...	1.1	20.3	3.5	T	0.30	0.15	0.11	5.5	63.4	3.0	0.22	0.41	1.9	0.08	0.9	0.64	1.57	2.5	8.6	1490	52.6	18.6	8.6	10.6	3.2	19.2
1562...	558...	1.4	22.5	2.3	T	0.24	0.12	0.05	5.1	64.5	1.8	0.44	0.27	1.5	0.15	0.9	0.45	2.22	9.2	19.8	1450	45.9	30.0	9.6	7.0	2.6	16.6
Average.....		1.5	21.2	3.2	0.01	0.26	0.10	0.19	5.2	63.2	2.7	0.36	0.39	1.8	0.17	0.7	0.60	1.75	5.3	13.1	1730	48.3	24.4	8.5	9.6	3.1	18.1
Group 6																											
1570...	573...	1.9	22.2	1.8	T	0.24	0.05	0.02	5.2	63.8	2.4	0.37	0.61	1.8	0.28	1.2	0.35	2.88	4.7	19.6	1470	43.3	31.1	10.7	5.5	3.1	16.2
1527...	453...	1.5	20.9	3.5	T	0.27	0.34	0.17	4.8	66.0	0.7	0.11	0.51	1.5	0.12	2.4	0.73	1.37	5.2	13.3	1500	58.4	15.9	6.8	10.6	2.6	17.4
1509...	399...	1.1	21.6	2.5	T	0.32	0.02	0.06	5.0	62.7	4.1	0.25	0.77	1.9	0.07	0.5	0.50	2.00	8.0	17.0	1500	46.3	27.1	9.0	7.6	3.2	16.6
1565...	567...	1.5	20.4	3.4	0.00	0.32	0.22	0.19	6.3	64.6	0.9	0.23	0.59	1.7	0.07	0.5	0.54	1.85	4.2	12.2	1470	53.7	18.0	10.9	10.3	2.9	21.2
1522...	438...	1.0	21.5	2.8	0.04	0.29	0.22	0.09	5.3	63.9	2.5	0.40	0.69	1.7	0.04	0.7	0.53	1.89	5.3	16.0	1440	49.3	24.6	9.3	8.5	2.9	17.8
1561...	555...	0.9	20.5	3.1	T	0.25	0.17	0.02	6.1	66.0	0.9	0.14	0.34	1.7	0.11	0.6	0.51	1.97	8.5	18.8	1620	60.0	13.6	10.9	9.4	2.9	20.3
1581...	599...	1.9	22.3	2.0	0.01	0.23	0.12	0.13	3.7	67.0	2.0	0.19	0.12	0.06	1.00	1.0	0.54	1.85	2.0	4.1	2470	71.1	10.4	6.4	6.1	0.1	12.5
1559...	549...	1.8	19.5	3.4	T	0.27	0.09	0.04	5.6	64.0	2.3	0.36	0.61	2.3	0.09	1.9	0.61	1.65	0.6	0.4	2780	55.5	14.1	9.1	10.3	3.9	19.4
1510...	402...	1.2	22.5	2.4	T	0.27	0.03	0.07	4.8	62.1	3.7	1.04	0.54	1.8	0.06	0.7	0.50	2.00	9.1	18.4	1530	37.9	36.0	8.6	7.3	3.1	15.9
1557...	465...	1.3	20.8	2.9	T	0.31	0.14	0.21	5.8	64.1	1.9	0.43	0.61	1.8	0.13	1.1	0.50	2.00	7.8	18.4	1420	50.0	22.0	10.4	8.8	3.1	19.2
Average.....		1.4	21.2	2.8	0.01	0.28	0.14	0.10	5.3	64.4	2.1	0.35	0.54	1.6	0.20	1.1	0.53	1.95	5.5	13.8	1720	52.6	21.3	9.2	8.4	2.8	17.6

[a] Value is for C_2F and is not included in average for C_3A.

[b] White cement. Ratio of Fe_2O_3 and Al_2O_3 not included in averages.

(*Continued on p. 994.*)

TABLE II.—*Continued.*

Series	Cement	Loss on Ignition	Oxide Analyses, per cent												Insoluble Residue	Free Lime CaO	Ratio		Percentage Retained on		Surface Area, sq. cm. per g.	Calculated Compounds, per cent					
			SiO_2	Fe_2O_3	FeO	TiO_2	P_2O_5	Mn_2O_3	Al_2O_3	CaO	MgO	Na_2O	K_2O	SO_3			Fe_2O_3 to Al_2O_3	Al_2O_3 to Fe_2O_3	Sieve No. 200	Sieve No. 325		C_3S	C_2S	C_3A	C_4AF	$CaSO_4$	Sum of C_3A and C_4AF
GROUP 7																											
1539...	501...	1.7	19.4	3.5	0.09	0.24	0.34	0.23	6.4	63.9	0.9	0.05	0.64	2.4	0.11	1.3	0.55	1.83	7.0	14.2	1630	52.4	16.1	11.0	10.6	4.1	21.6
1538...	492...	1.2	22.4	1.9	T	0.27	0.03	0.05	4.9	65.4	1.3	0.27	0.76	1.5	0.14	0.9	0.39	2.58	5.5	13.9	1650	52.2	24.9	9.8	5.8	2.6	15.6
1591...	619...	2.1	22.3	2.0	0.01	0.26	0.09	0.03	4.9	62.8	2.6	0.52	0.50	1.9	0.13	0.5	0.41	2.45	9.5	19.5	1380	42.7	31.8	9.6	6.1	3.2	15.7
1529...	459...	2.0	20.5	3.4	T	0.27	0.31	0.16	4.7	65.5	0.7	0.25	0.47	1.6	0.14	2.4	0.72	1.38	2.3	3.1	2040	59.9	13.6	6.7	10.3	2.7	17.0
1517...	423...	0.9	22.3	2.0	T	0.23	0.13	0.09	4.6	62.7	4.7	0.30	0.45	1.7	0.08	1.0	0.44	2.30	6.7	14.9	1590	42.9	31.7	8.8	6.1	2.9	14.7
1540...	495...	1.0	20.4	3.2	T	0.24	0.19	0.24	5.7	64.1	2.4	0.42	0.60	1.7	0.07	1.5	0.56	1.78	6.5	17.2	1480	51.9	19.4	9.7	9.7	2.9	19.4
1533...	477...	1.4	21.0	2.2	T	0.26	0.19	0.21	5.8	65.6	1.4	0.00	0.35	1.7	0.18	1.4	0.38	2.63	9.6	20.4	1650	54.6	19.1	11.9	6.7	2.9	18.6
1597...	629...	1.4	19.0	2.4	0.03	0.27	0.27	0.09	5.9	64.0	3.4	0.23	0.53	2.6	0.06	0.8	0.41	2.46	0.1	3.3	2050	62.3	7.6	11.6	7.3	4.4	19.9
1526...	450...	1.7	19.5	3.3	T	0.26	0.14	0.14	4.4	65.3	3.0	0.24	0.11	2.2	0.05	1.7	0.75	1.33	3.2	5.4	2460	70.0	3.2	6.1	10.0	3.7	16.1
1556...	552...	1.9	19.4	3.5	0.00	0.28	0.11	0.11	5.4	63.8	2.9	0.21	0.37	2.1	0.16	1.3	0.65	1.54	0.3	1.4	2820	59.6	10.7	8.4	10.6	3.6	19.0
Average.....		1.5	20.6	2.7	0.01	0.26	0.18	0.14	5.3	64.3	2.3	0.25	0.48	1.9	0.11	1.3	0.53	2.03	5.1	11.3	1880	54.9	17.8	9.4	8.3	3.3	17.8
GROUP 8																											
1516...	420...	1.3	20.9	2.1	T	0.29	T	0.04	5.6	64.2	3.1	0.21	0.39	2.2	0.11	0.8	0.38	2.66	5.1	9.0	2480	52.1	20.7	11.3	6.4	3.7	17.7
1626...	663...	1.4	21.9	2.2	0.04	0.26	0.21	0.06	5.2	63.7	2.9	0.21	0.37	1.7	0.11	1.0	0.42	2.36	3.5	11.6	1730	45.7	28.4	10.0	6.7	2.9	16.7
1525...	447...	1.3	20.2	3.4	T	0.31	0.15	0.12	5.2	65.0	2.7	0.26	0.30	1.4	0.02	1.4	0.65	1.53	4.2	13.0	1600	61.5	11.6	8.0	10.3	2.4	18.3
1632...	669...	1.4	20.2	2.4	0.07	0.26	0.29	0.07	6.0	63.5	3.7	0.29	0.45	1.9	0.24	0.4	0.40	2.50	5.1	11.1	1760	54.1	17.2	11.8	7.3	3.2	19.1
1588...	615...	2.3	22.2	0.3	0.05	0.31	0.00	T	5.5	64.8	2.8	0.00	0.12	1.6	0.10	1.4	*b*	*b*	3.6	11.6	1920	47.2	28.1	14.1	0.9	2.7	15.0
1592...	620...	1.3	21.7	2.2	0.03	0.29	0.09	0.05	5.7	63.0	3.4	0.15	0.71	1.7	0.04	0.3	0.39	2.59	0.9	19.2	1490	43.8	29.2	11.4	6.7	2.9	18.1
1590...	618...	1.2	20.3	2.8	0.16	0.34	0.06	0.08	6.9	63.2	2.7	0.17	0.41	1.8	0.08	0.3	0.41	2.46	5.5	12.5	1810	46.1	23.5	13.5	8.5	3.1	22.0
1595...	627...	1.9	23.3	2.0	0.00	0.26	0.05	0.05	4.8	64.4	1.2	0.46	0.53	1.4	0.07	2.0	0.42	2.40	2.3	10.4	1810	37.6	38.5	9.3	6.1	2.4	15.4
1528...	456...	2.3	20.7	3.5	T	0.26	0.31	0.17	4.8	65.1	0.7	0.29	0.54	1.5	0.13	2.4	0.73	1.37	2.9	3.2	2480	56.3	17.0	6.8	10.6	2.6	17.4
1542...	504...	1.2	22.0	2.4	T	0.19	0.08	0.04	5.3	62.6	4.0	0.04	0.48	1.7	0.11	1.1	0.45	2.21	4.5	13.1	1470	39.1	33.6	10.0	7.3	2.9	17.3
Average.....		1.6	21.3	2.3	0.04	0.28	0.12	0.07	5.5	64.0	2.7	0.21	0.43	1.7	0.10	1.1	0.47	2.23	3.8	11.5	1860	48.4	23.8	10.6	7.1	2.9	17.7
GROUP 9																											
1618...	645...	2.0	20.0	3.1	0.45	0.24	0.06	0.03	5.2	63.0	3.1	0.29	0.61	1.9	0.14	2.2	0.60	1.68	5.5	11.3	2060	50.6	19.3	8.5	9.4	3.2	17.9
1609...	641...	0.9	22.1	2.3	0.00	0.23	0.15	T	5.4	65.2	0.9	0.39	0.66	1.8	0.13	0.6	0.43	2.34	4.6	11.2	1720	50.1	25.6	10.4	7.0	3.1	17.4
1623...	656...	1.4	20.8	2.4	0.01	0.29	0.03	0.06	5.5	63.1	3.7	0.04	0.90	1.8	0.19	1.4	0.44	2.29	8.5	17.6	1620	47.4	23.9	10.5	7.3	3.1	17.8
1582...	602...	1.1	21.1	2.6	0.01	0.33	0.11	0.03	5.4	63.1	3.4	0.26	0.94	1.9	0.16	0.8	0.48	2.08	8.0	17.2	1400	47.6	24.6	9.9	7.9	3.2	17.8
1621...	650...	1.4	21.5	2.4	0.00	0.25	0.02	0.03	5.7	64.1	2.3	0.14	0.57	1.6	0.07	2.0	0.42	2.38	12.0	23.0	1580	43.0	29.3	11.0	7.3	2.7	18.3
1573...	594...	2.0	23.6	2.1	0.12	0.28	0.17	T	6.2	61.3	1.3	0.33	0.43	2.6	2.60	0.9	0.34	2.95	6.8	17.6	1710	14.2	57.0	12.9	6.4	4.4	19.3
1617...	644...	1.5	20.5	2.5	0.52	0.24	0.06	0.03	5.2	63.2	3.2	0.24	0.55	1.8	0.19	1.6	0.48	2.08	6.1	14.4	1720	51.1	20.3	9.5	7.6	3.1	17.1
1500...	372...	1.6	21.3	2.6	T	0.23	0.03	0.08	6.4	64.4	1.4	0.12	0.64	1.5	0.25	1.0	0.41	2.46	14.2	23.3	1480	45.0	27.2	12.6	7.9	2.6	20.5
1575...	579...	1.5	21.1	2.1	0.10	0.27	0.12	0.03	5.7	64.6	2.3	0.44	0.15	1.7	0.10	0.9	0.37	2.72	3.2	13.7	1540	52.6	20.9	11.6	6.4	2.9	18.0
1560...	537...	1.4	20.1	3.6	0.09	0.31	0.12	0.06	6.2	63.1	2.0	0.72	0.47	1.9	0.10	0.9	0.58	1.72	10.3	20.0	1550	48.0	21.5	10.3	10.9	3.2	21.2
Average.....		1.5	21.2	2.6	0.13	0.27	0.09	0.04	5.7	63.5	2.4	0.30	0.59	1.9	0.39	1.2	0.46	2.27	7.9	16.9	1640	45.0	27.0	10.7	7.8	3.2	18.5

Group 10

1641...	685...	1.3	22.0	2.4	0.03	0.25	0.13	0.05	5.4	64.9	0.8	0.46	0.57	1.9	0.24	0.8	0.44	2.25	5.8	13.1	1530	48.4	26.6	10.2	7.3	3.2	17.5
1574...	595...	1.9	21.9	2.1	0.19	0.27	0.15	T	6.1	63.8	1.3	0.34	0.50	1.6	0.07	1.5	0.34	2.90	6.3	19.5	1490	38.4	33.9	12.6	6.4	2.7	19.0
1513...	411...	0.6	21.2	2.5	0.02	0.30	T	0.65	6.1	64.3	2.2	0.16	0.25	1.9	0.08	0.5	0.41	2.44	10.6	22.2	1450	48.5	24.3	11.9	7.6	3.2	19.5
1501...	375...	1.3	20.8	2.3	T	0.27	0.04	0.03	5.6	64.4	2.8	0.35	0.44	1.8	0.17	1.5	0.41	2.43	5.9	15.5	1580	51.7	20.7	10.9	7.0	3.1	17.9
1631...	668...	0.9	21.9	2.2	0.02	0.21	0.21	0.03	5.0	63.3	4.2	0.38	0.35	1.6	0.10	0.9	0.44	2.27	7.7	17.6	1470	46.1	28.1	9.5	6.7	2.7	16.2
1534...	480...	1.0	19.8	2.9	0.03	0.28	0.15	0.14	6.9	65.0	1.4	0.10	0.40	1.8	0.13	0.5	0.42	2.38	5.9	15.0	1720	56.3	14.4	13.4	8.8	3.1	22.2
1544...	522...	1.7	21.2	2.2	0.05	0.28	0.05	0.11	5.8	65.7	1.1	0.33	0.23	1.5	0.17	1.4	0.38	2.64	7.1	14.3	1790	54.1	20.1	11.6	6.7	2.6	18.3
1515...	417...	1.2	21.1	2.1	T	0.31	T	0.03	5.5	64.6	2.9	0.17	0.50	1.7	0.11	1.4	0.38	2.62	7.4	14.9	1730	51.9	21.4	11.0	6.4	2.9	17.4
1600...	632...	1.4	21.5	2.5	0.00	0.29	0.03	0.16	5.9	64.6	1.1	0.46	0.46	1.8	0.10	1.1	0.42	2.36	7.0	15.4	1370	46.5	26.6	11.4	7.6	3.1	19.0
1519...	429...	2.0	20.1	2.8	0.25	0.30	0.21	0.12	6.8	63.9	1.3	0.06	0.63	1.9	0.30	1.4	0.41	2.43	5.1	12.7	1810	46.4	22.7	13.3	8.5	3.2	21.8
Average		1.2	21.2	2.4	0.06	0.28	0.10	0.13	5.9	64.5	1.9	0.28	0.43	1.8	0.15	1.1	0.41	2.47	6.9	16.0	1590	48.8	23.9	11.6	7.2	3.0	18.8

Group 11

1572...	593...	1.7	21.5	2.2	0.07	0.30	0.19	T	6.6	64.3	1.4	0.20	0.37	1.7	0.08	1.0	0.33	3.00	7.4	18.4	1690	41.8	30.2	13.8	6.7	2.9	20.5
1530...	468...	1.6	20.5	2.3	T	0.26	0.11	0.05	6.0	62.8	3.1	0.00	1.41	1.9	0.14	1.7	0.38	2.61	8.2	17.6	1550	43.7	25.9	12.0	7.0	3.2	19.0
1615...	680...	1.3	19.8	2.2	0.15	0.29	0.36	0.10	6.5	63.7	3.1	0.40	0.59	1.8	0.15	0.9	0.34	2.95	9.7	20.1	1290	53.1	16.8	13.5	6.7	3.1	20.2
1598...	630...	1.9	21.5	2.2	0.01	0.23	0.00	0.04	5.5	64.0	2.3	0.30	0.62	1.5	0.08	1.9	0.40	2.50	15.7	25.6	1200	44.8	27.9	10.8	6.7	2.6	17.5
1625...	662...	1.6	19.5	2.7	0.18	0.37	0.05	T	7.1	63.3	2.8	0.04	0.66	1.8	0.22	1.7	0.38	2.63	4.3	14.7	1310	45.7	21.5	14.2	8.2	3.1	22.4
1535...	483...	1.2	21.5	2.3	0.10	0.31	0.04	0.06	5.9	61.9	3.7	0.25	0.99	1.8	0.32	0.9	0.39	2.56	7.8	17.2	1550	36.7	34.1	11.7	7.0	3.1	18.7
Average		1.6	20.7	2.3	0.09	0.29	0.13	0.04	6.3	63.3	3.1	0.20	0.77	1.8	0.17	1.4	0.37	2.71	8.9	18.9	1430	44.3	26.1	12.7	7.1	3.0	19.8

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[b] White cement. Ratio of Fe_2O_3 and Al_2O_3 not included in averages.

TABLE III.—SUMMARY OF THE ANALYSES OF THE 106 COMMERCIAL CEMENTS COMPRISING THE 11 GROUPS OF TABLES I AND II.[a]
Groups are numbered in descending order of sulfate resistance

Analytical Data		Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9	Group 10	Group 11
OXIDE ANALYSES, PER CENT												
Loss on ignition	Average	1.4	1.7	1.6	1.8	1.5	1.4	1.5	1.6	1.5	1.2	1.6
	Maximum	2.2	4.1	2.6	3.0	2.1	1.9	2.1	2.3	2.0	2.0	1.9
	Minimum	1.1	0.9	0.6	1.1	1.1	0.9	0.9	1.2	0.9	0.6	1.2
SiO_2	Average	22.0	21.9	21.4	21.0	21.2	21.2	20.6	21.3	21.2	21.2	20.7
	Maximum	23.6	22.9	23.1	23.6	22.5	22.5	22.4	23.3	23.6	22.0	21.5
	Minimum	19.9	20.8	19.8	19.0	20.0	19.5	19.0	20.2	20.0	19.8	19.5
Fe_2O_3	Average	4.0	3.8	3.2	2.9	3.2	2.8	2.7	2.3	2.6	2.4	2.3
	Maximum	5.2	5.4	6.0	5.0	5.5	3.5	3.5	3.5	3.6	2.9	2.7
	Minimum	2.0	2.6	2.0	0.4	2.3	1.8	1.9	0.3	2.1	2.1	2.2
FeO	Average	0.04	0.03	0.02	0.02	0.01	0.01	0.01	0.04	0.13	0.06	0.09
	Maximum	0.30	0.25	0.07	0.16	0.05	0.04	0.09	0.16	0.52	0.25	0.18
	Minimum	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	T
TiO_2	Average	0.27	0.24	0.24	0.31	0.26	0.28	0.26	0.28	0.27	0.28	0.29
	Maximum	0.63	0.34	0.30	0.64	0.33	0.32	0.28	0.34	0.33	0.31	0.37
	Minimum	0.19	0.15	0.19	0.22	0.19	0.23	0.23	0.19	0.23	0.21	0.23
P_2O_5	Average	0.14	0.10	0.09	0.14	0.10	0.14	0.18	0.12	0.09	0.10	0.13
	Maximum	0.29	0.37	0.20	0.29	0.24	0.34	0.34	0.31	0.17	0.21	0.36
	Minimum	0.06	0.03	0.02	0.00	T	0.02	0.03	0.00	0.02	T	0.00
Mn_2O_3	Average	0.11	0.06	0.07	0.12	0.19	0.10	0.14	0.07	0.04	0.13	0.04
	Maximum	0.47	0.10	0.11	0.30	0.69	0.21	0.24	0.17	0.08	0.65	0.10
	Minimum	0.01	0.03	0.04	T	0.04	0.02	0.03	T	T	T	T
Al_2O_3	Average	4.1	4.2	4.3	4.6	5.2	5.3	5.3	5.5	5.7	5.9	6.3
	Maximum	4.9	5.3	4.7	6.2	5.8	6.3	6.4	6.9	6.4	6.9	7.1
	Minimum	2.8	3.4	3.7	3.6	4.5	3.7	4.4	4.8	5.2	5.0	5.5
CaO	Average	63.9	62.8	63.6	64.2	63.2	64.4	64.3	64.0	63.5	64.5	63.3
	Maximum	65.9	65.5	66.2	66.3	64.5	67.0	65.6	65.1	65.2	65.7	64.3
	Minimum	62.1	58.2	59.9	61.4	61.3	62.1	62.7	62.6	61.3	63.3	61.9
MgO	Average	2.0	2.8	3.1	2.1	2.7	2.1	2.3	2.7	2.4	1.9	3.1
	Maximum	2.6	4.1	5.4	3.0	4.6	4.1	4.7	4.0	3.7	4.2	3.7
	Minimum	1.2	1.1	1.3	1.0	1.7	0.7	0.7	0.7	0.9	0.8	1.4
Na_2O	Average	0.39	0.25	0.25	0.19	0.36	0.35	0.25	0.21	0.30	0.28	0.20
	Maximum	1.20	0.44	0.53	0.48	1.20	1.04	0.52	0.46	0.72	0.46	0.40
	Minimum	0.15	0.11	0.00	0.00	0.10	0.11	0.00	0.00	0.04	0.06	0.00
K_2O	Average	0.29	0.47	0.42	0.48	0.39	0.54	0.48	0.43	0.59	0.43	0.77
	Maximum	0.51	0.73	0.75	1.22	0.77	0.77	0.76	0.71	0.94	0.63	1.41
	Minimum	0.02	0.18	0.12	0.20	0.02	0.12	0.11	0.12	0.15	0.23	0.37
SO_3	Average	1.7	1.9	1.8	1.9	1.8	1.6	1.9	1.7	1.9	1.8	1.8
	Maximum	1.9	2.3	2.5	2.4	2.5	2.3	2.6	2.2	2.6	1.9	1.9
	Minimum	1.4	1.6	1.3	1.5	1.5	0.06	1.5	1.4	1.5	1.5	1.5
Insoluble residue	Average	0.15	0.15	0.16	0.17	0.17	0.20	0.11	0.10	0.39	0.15	0.17
	Maximum	0.28	0.36	0.35	0.42	0.26	1.0	0.18	0.24	2.60	0.30	0.32
	Minimum	0.06	0.05	0.06	0.08	0.08	0.04	0.05	0.02	0.07	0.07	0.08
Free lime CaO	Average	0.7	0.7	0.8	1.3	0.7	1.1	1.3	1.1	1.2	1.1	1.4
	Maximum	1.3	1.4	1.8	3.0	1.1	2.4	2.4	2.4	2.2	1.5	1.9
	Minimum	0.1	0.1	0.1	0.4	0.2	0.5	0.5	0.3	0.6	0.5	0.9
IRON-ALUMINA RATIOS												
Ratio of Fe_2O_3 to Al_2O_3	Average	1.01	0.91	0.75	0.60	0.60	0.53	0.53	0.47	0.46	0.41	0.37
	Maximum	1.86	1.23	1.46	1.00	0.95	0.73	0.75	0.73	0.60	0.44	0.40
	Minimum	0.53	0.49	0.47	0.44	0.45	0.35	0.38	0.38	0.34	0.34	0.33
Ratio of Al_2O_3 to Fe_2O_3	Average	1.09	1.19	1.48	1.75	1.75	1.95	2.03	2.23	2.27	2.47	2.71
	Maximum	1.90	2.04	2.14	2.28	2.22	2.88	2.63	2.66	2.95	2.90	3.00
	Minimum	0.54	0.82	0.68	1.00	1.05	1.37	1.33	1.37	1.68	2.25	2.50

[a] T = a trace.

TABLE III.—*Continued.*

Analytical Data		Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8	Group 9	Group 10	Group 11
FINENESS DETERMINATIONS												
Per cent retained on sieve No. 200	Average	6.7	4.0	4.8	3.8	5.3	5.5	5.1	3.8	7.9	6.9	8.9
	Maximum	12.5	7.5	8.4	9.7	9.2	9.1	9.6	5.5	14.2	10.6	15.7
	Minimum	2.6	2.0	1.4	0.3	1.0	0.6	0.1	0.9	3.2	5.1	4.3
Per cent retained on sieve No. 325	Average	16.5	10.6	11.5	10.2	13.1	13.8	11.3	11.5	16.9	16.0	18.9
	Maximum	30.0	16.8	16.9	19.4	19.8	19.6	20.4	19.2	23.3	22.2	25.6
	Minimum	8.0	5.8	3.9	0.6	1.3	0.4	1.4	3.2	11.2	12.7	14.7
Surface area, sq. cm. per g.	Average	1510	1790	1850	1850	1730	1720	1880	1860	1640	1590	1430
	Maximum	1750	2250	2560	2360	2600	2780	2820	2480	2060	1810	1690
	Minimum	1160	1520	1480	1540	1430	1420	1380	1470	1400	1370	1200
CALCULATED COMPOUNDS, PER CENT												
C_3S	Average	52.1	47.8	54.4	52.9	48.3	52.6	54.9	48.4	45.0	48.8	44.3
	Maximum	62.5	55.3	67.9	67.5	54.6	71.1	70.0	61.5	52.6	56.3	53.1
	Minimum	36.4	26.8	44.8	43.1	43.4	37.9	42.7	37.6	14.2	38.4	36.7
C_2S	Average	23.7	26.7	20.4	20.4	24.4	21.3	17.8	23.8	27.0	23.9	26.1
	Maximum	39.1	42.9	31.9	31.8	30.7	36.0	31.8	38.5	57.0	33.9	34.1
	Minimum	11.7	18.9	5.6	6.0	18.6	10.4	3.2	11.6	19.3	14.4	16.8
C_3A	Average	4.5	4.8	6.0	8.6	8.5	9.2	9.4	10.6	10.7	11.6	12.7
	Maximum	6.7	9.7	8.7	10.3	9.7	10.9	11.9	14.1	12.9	13.4	14.2
	Minimum	3.3	2.2	0.7	4.8	6.0	6.4	6.1	6.8	8.5	9.5	10.8
C_4AF	Average	12.0	11.4	9.7	8.7	9.6	8.4	8.3	7.1	7.8	7.2	7.1
	Maximum	15.8	16.4	18.2	15.2	16.7	10.6	10.6	10.6	10.9	8.8	8.2
	Minimum	6.1	7.9	6.1	1.2	7.0	5.5	5.8	0.9	6.4	6.4	6.7
$CaSO_4$	Average	2.9	3.2	3.0	3.2	3.1	2.8	3.3	2.9	3.2	3.0	3.0
	Maximum	3.2	3.9	4.3	4.1	4.3	3.9	4.4	3.7	4.4	3.2	3.2
	Minimum	2.4	2.7	2.2	2.6	2.6	0.1	2.6	2.4	2.6	2.6	2.6
Sum of C_3A and C_4AF	Average	16.5	16.2	15.7	17.3	18.1	17.6	17.8	17.7	18.5	18.8	19.8
	Maximum	19.9	18.9	18.9	21.2	22.7	21.2	21.6	22.0	21.2	22.2	22.4
	Minimum	12.8	13.7	12.8	10.1	15.6	12.5	14.7	15.0	17.1	16.2	17.5

cent $MgSO_4$ solutions, (2) length increases of the cylinders in the 1 per cent Na_2SO_4, and (3) strength ratios of the Medicine Lake cylinders. The cements were rated from best to poorest, from 1 to 106, under each test and the ratings for each cement totaled; therefore, the cement with the lowest total scored highest in resistance. An admitted weakness of the work is the fact that only one or two lots of each cement were tested. On the other hand, numerous cements closely comparable in chemical composition were tested so that the lack of repeat tests of individual cements is fairly well compensated so far as trends are concerned.

Explanations Regarding Tabulated Data.—In Table I are recorded results of the compression tests of the Medicine Lake cylinders and also the expansion tests of the cylinders in the laboratory solutions. In Table II are recorded the oxide analyses, calculated compounds, and fineness determinations of the cements while the essential data of Table II are summarized in Table III. The 106 cements in the eleven groups of the tables are arranged in descending order of resistance, as determined. Chemical analyses and fineness determinations of these cements were made in 1935 to 1937 for the Bureau of Agricultural Engineering of the U. S. Department of Agriculture by the National Bureau of Standards, which reported as follows regarding details of the analyses:

Ferrous Oxide (FeO).—"Determined by dissolving the sample in hydrochloric

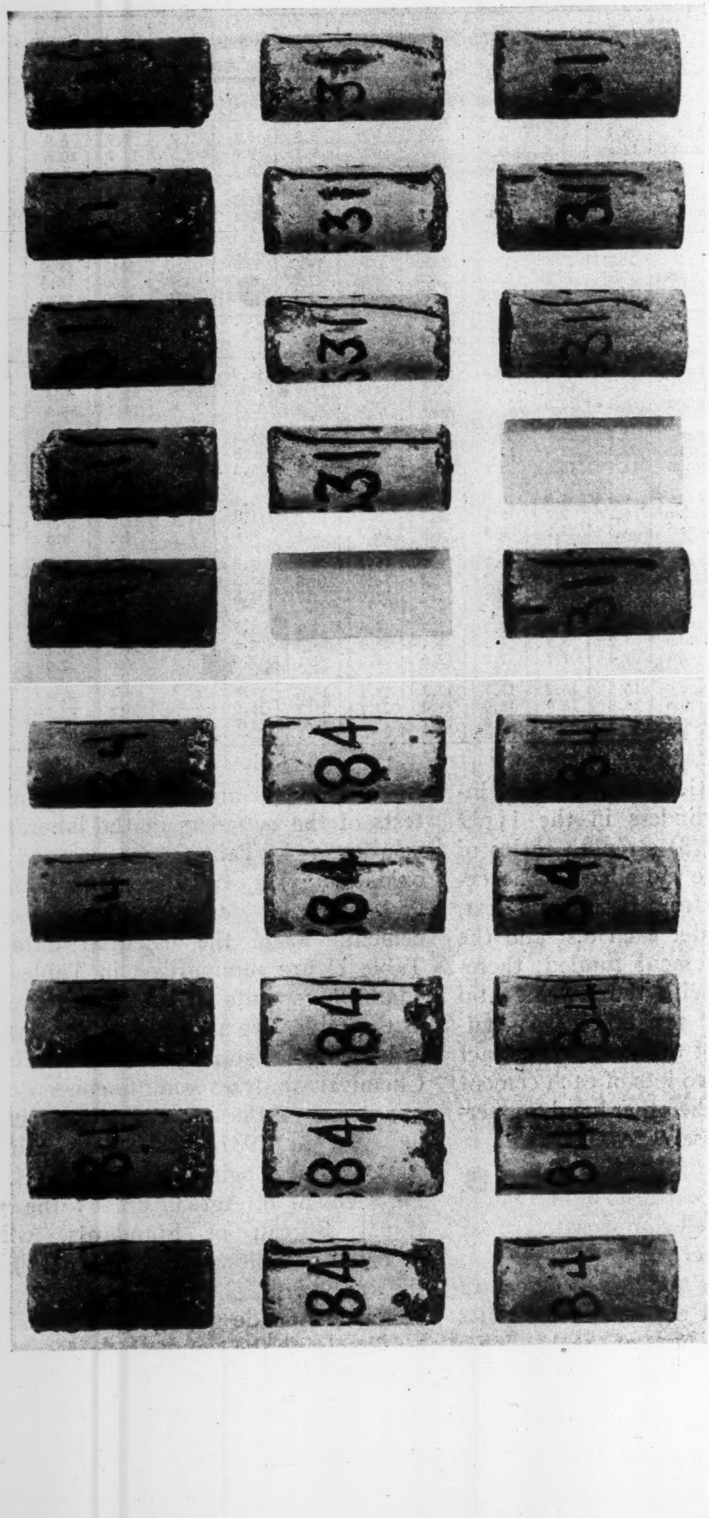

Fig. 1.—Condition of Cylinders in Two Series After Exposure for 5 yr. as Follows:
Upper row in natural sulfate water of Medicine Lake.
Middle row in 1 per cent laboratory solutions of $MgSO_4$.
Lower row in 1 per cent laboratory solutions of Na_2SO_4.

As rated for resistance, 38 cements separated cylinders of Series 1584 and 1531.
Missing cylinders of Series 1531 had completely failed.

acid in an atmosphere of carbon dioxide and titrating with dichromate."

Free Lime.—"Determined by the method of Lerch and Bogue modified by the addition of barium chloride."

Percentage Retained on No. 325 sieve. —"Determined by wet method as prescribed in Wagner Turbidimeter Procedure."

Surface Areas.—"Determined with the Wagner Turbidimeter."

The compounds of the cements were computed as outlined by R. H. Bogue in *Paper No. 21*, "Calculations of Compounds in Portland Cement," issued by the Portland Cement Association Fellowship at the National Bureau of Standards. Throughout this report the cement compounds are designated by commonly accepted abbreviations as follows:

Compound	Abbreviation
Tricalcium silicate	C_3S
Dicalcium silicate	C_2S
Tricalcium aluminate	C_3A
Tetracalcium alumino-ferrite	C_4AF
Dicalcium ferrite	C_2F

A wide difference in resistance to sulfates was displayed by the 106 cements. It is not possible, at this time, to express the difference in ratios as the tests have not continued long enough in the case of the more resistant cements. However, under the conditions imposed, the cements of group 1 of Table I were definitely five or six times as resistant as those of group 11, while indications warrant the assumption that ultimately this difference will be ten or twelve times. Figure 1 illustrates the general uniformity of results obtained by the three conditions of exposure and also shows that the sulfate resistance, as determined, is further evidenced by the appearances of the specimens. It will be seen that the cylinders of series 1584 show only slight action while the Medicine Lake cylinders of series 1531 are definitely deteriorating, with one cylinder already gone from each of the laboratory solutions. Referring to either Table I or Table II it will be noted that 38 cements separate those two, as rated. These photographs were selected as fairly representing the visual gradation at 5 yr. of the cylinders of groups 1, 2, 3, and 4.

TABLE IV.—NUMBER OF CEMENTS IN EACH OF THE ELEVEN GROUPS OF TABLE II WHICH WOULD MEET SPECIFICATIONS WITH A MAXIMUM LIMIT OF ALUMINUM OXIDE OR TRICALCIUM ALUMINATE AS INDICATED.

Group	Aluminum Oxide							Tricalcium Aluminate													
	3.5 %	4.0 %	4.5 %	5.0 %	5.5 %	6.0 %	6.5 %	3.5 %	4.0 %	4.5 %	5.0 %	5.5 %	6.0 %	6.5 %	7.0 %	7.5 %	8.0 %	8.5 %	9.0 %	9.5 %	10.0 %
1	1	5	8	10	10	10	10	3	5	6	6	9	9	9	10	10	10	10	10	10	10
2	1	4	8	9	10	10	10	3	4	5	6	7	8	9	9	9	9	9	9	9	10
3	0	2	6	10	10	10	10	1	1	2	2	3	4	4	6	9	9	9	10	10	10
4	0	1	1	4	8	9	10			0	1	1	1	1	1	3	3	3	5	6	9
5		0	1	3	8	10	10					0	2	2	2	2	2	3	6	7	10
6	0	1	1	4	6	8	10						0	1	2	2	2	2	4	6	6
7		0	1	5	6	9	10						0	1	2	2	2	3	4	4	7
8			0	2	6	9	9							0	1	1	2	2	2	3	5
9				0	5	7	10										0	1	1	2	3
10			0	1	3	6	8												0	1	1
11				0	1	3	4														0
Total	2	13	26	48	73	91	101	7	10	13	15	20	24	27	33	38	39	42	51	58	71

CEMENT COMPOSITION AND SULFATE RESISTANCE

The analytical data of Table II have been summarized in Tables III and IV for greater convenience of study. The data of Tables III and IV reveal a few significant trends.

1. Aluminum Oxide (Al_2O_3).—There were 26 of the 106 cements in which Al_2O_3 did not exceed 4.5 per cent. Of those 26 cements, 22 were among the 30 most resistant of the 106 and none were among the 37 least resistant. The percentage of this oxide increased consistently from an average of 4.1 per cent for group 1 to 6.3 per cent for group 11.

2. Tricalcium Aluminate ($3CaO \cdot Al_2O_3$) or as Abbreviated (C_3A).—There were 20 of the 106 cements in which this calculated compound did not exceed 5.5 per cent. All 20 of these cements were among the 40 most resistant of the 106 while 16 were among the top 20. The average percentage of this calculated compound increased consistently from 4.5 per cent for group 1 to 12.7 per cent for group 11.

3. The ratio of Fe_2O_3 to Al_2O_3 averaged close to 1.00 for the 20 most resistant of the 106 cements. This ratio decreased consistently in groups 3 to 11, reaching a low of 0.37 in group 11.

4. Tetracalcium Alumino-Ferrite ($4CaO \cdot Al_2O_3 \cdot Fe_2O_3$) or as Abbreviated ($C_4AF$).—This calculated compound generally decreased as resistance decreased, averaging 12.0 per cent for group 1 and 7.1 per cent for group 11. The sum of C_3A plus C_4AF varied but slightly for the eleven groups, the range being between a low of 15.7 per cent for group 3 to a high of 19.8 for group 11.

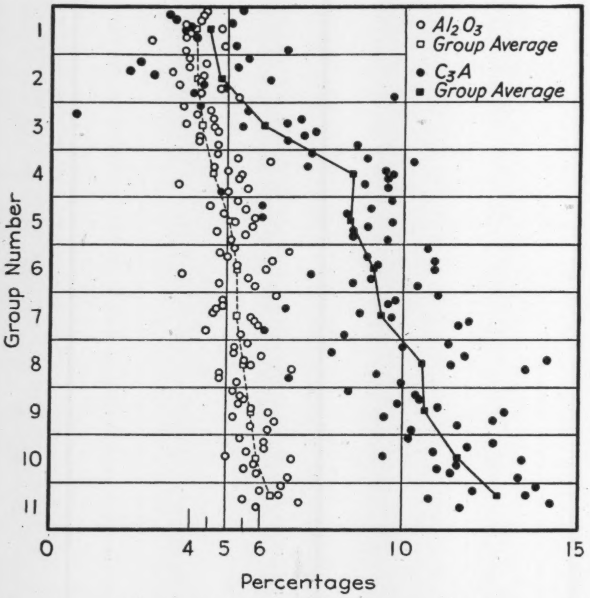

FIG. 2.—One Hundred Six Commercial Cements of the Eleven Groups of Table I and II, Arranged in Descending Order of Sulfate Resistance, Plotted Against Aluminum Oxide and Tricalcium Aluminate Percentages.

Reviewing all the data here presented, it is evident that a close relationship exists between the sulfate resistance of these commercial portland cements and the aluminum oxide (Al_2O_3) content, however considered. In Fig. 2 the cements of the eleven groups are plotted against both Al_2O_3 and the calculated compound C_3A. These graphs clearly show that the relationship to resistance is essentially similar for 4.5 per cent Al_2O_3 and 5.5 per cent C_3A.

Study of the data of Table IV reveals that a specification limit of 4.5 per cent for Al_2O_3 would have passed 8 cements each of groups 1 and 2, 6 cements of group 3, and 1 cement each of groups 4, 5, 6, and 7, a total of 26 cements. A limit of 5.5 per cent for the calculated compound C_3A would have passed 9 cements of group 1, 7 cements of group 2, 3 cements of group 3, and 1 cement of group 4, a total of 20 cements, while all 66 cements of groups 5 to 11, inclusive, would have been eliminated.

The evidence, as indicated by the tests and analyses of these 106 cements, clearly favors use of cements low in the calculated compound C_3A for sulfate resistance. As a matter of fact, there were only 3 cements of the entire 106 that fell very far out of line. One of these is cement 516 of series 1547, with 9.7 per cent C_3A, rated last in group 2. Nothing in the chemical analyses reveals why this cement rated as high in resistance as it did. Another cement out of line is 513 of series 1546, with 0.07 per cent C_3A, rated third in group 3. This cement carried 5.4 per cent MgO which probably accounts for the relatively poor showing in the laboratory solutions, as rated by expansion measurements, although the 5-yr. strengths of the Medicine Lake cylinders were fairly high. The third cement out of line is 471 of series 1531, with 4.8 per cent C_3A, rated last in group 4. This cement was the second highest of the 106 cements in K_2O, with 1.22 per cent.

CONCLUSIONS

Such studies of the analytical data as have been made for this progress report do not reveal that the alkalies ($NaO + K_2O$), free lime, magnesium oxide, or the several oxides not ordinarily considered in a cement analysis, within the limits of occurrence in the 106 cements, greatly affected resistance, nor did fineness of grinding. Any possible influence one or more of the minor oxides may have had on resistance is obscured by the greater effect of variations in the quantity of alumina. However, the data need to be explored further along this line.

No consideration, whatever, has been given to variables of manufacturing practices at the different mills. Here again, this factor seems to have been overshadowed by the greater influence exerted by variations in the quantity of alumina.

Judged by results of the tests as applied to the 106 commercial cements of this paper, a specified upper limit of 5.5 per cent tricalcium aluminate would come as near to securing cements of high resistance to attack by magnesium and sodium sulfate as reasonably could be expected and, at the same time, would eliminate all cements of low resistance. It appears sound, therefore, to rely chiefly on this chemical approach for the selection of a cement to be exposed to the action of these sulfates, until a short-time laboratory test may be devised that is of proven greater reliability.

DISCUSSION

THE CHAIRMAN (*Mr. M. A. Swayze*[1]).—Referring to Mr. Miller's diagram showing the Relation of C_3A to durability, one might point out that compound composition is not the whole answer, and that there must be something more to the effect of processing than purely chemical composition alone. One other point that is extremely interesting is the fact that Mr. Miller's sulfate-resistant cements—those in group 1 and possibly in group 2—were on the average high in tricalcium silicate, which is directly the opposite to what Committee C-1 on Cement has recommended as a sulfate-resisting cement. It shows that the committee probably will wish to review Mr. Miller's work very carefully and give some consideration as to whether to continue to keep the sulfate-resisting cement in the low heat - low C_3S category.

MR. P. H. BATES.[2]—I should like to say a few words about the diagram which Mr. Miller has presented. Those in Committee C-1 who know me, know that I very much protest this matter of a study of trends and averages to the exclusion of the exceptions. I think I have cited the fact to the members of the committee that if we were average human beings, we would not be here. We are exceptions, hence we are here. After the Kentucky Derby has been run and Pimlico, etc., is the average horse bought for breeding purposes? No, for that purpose interest is not in the average horse at all, but in the exception—the horse that won.

Here in the diagram you can note the trend, but please note the exceptions. Note that there are cements of the same aluminate content but vastly different physical properties; also that some of the cements have the same physical property but distinctly different aluminate content. Note particularly that one of these cements gave excellent resistance but is much beyond the aluminate limit suggested by Mr. Miller.

Just as in plant development, the average plant is neglected in study but attention is directed to the "sport," so in cement development and study the same should hold. This cement with the high aluminate content but excellent resistance is, according to the trend, a "sport" cement. It is the one which deserves attention. It proves that, if in this investigation the cement composition was the only variable, it is possible to make a high aluminate cement having excellent sulfate resistance. Those who are interested in the production of cements and those studying cements for the purpose of preparing standards should always study the trends only to find the exceptions, then study the unusual good exceptions.

MR. DALTON G. MILLER.[3]—You all know the old saying that the difference between an optimist and a pessimist is that one sees the doughnut and the other sees the hole. I am going to look at the doughnut even if Mr. Bates is going to look at the hole. I do not believe there can be any chemical specification for a sulfate-resistant cement that will ever be as good as a short-time physical test, or possibly a combination of physical and chemical tests. Up to the present time, no satisfactory short-time test has been devised and until that is done chief reliance must be on chemical composition. There was not bad correlation between the behavior of these cements and chemical composition; in fact, the correlation between sulfate resistance, as determined, and chemical composition is remarkably close. It is true that there are a few exceptions and unquestionably the exceptions merit careful study as to the causes for the exceptions.

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[1] Director of Research, Lone Star Cement Corp., Hudson, N. Y.

[2] Chief, Clay and Silicate Products Division, National Bureau of Standards, Washington, D. C.

[3] Senior Drainage Engineer, Division of Drainage, Soil Conservation, U. S. Department of Agriculture, University Farm, St. Paul, Minn.

MR. F. O. ANDEREGG.[4]—Two factors might be mentioned to explain some of the exceptions noted in this paper: the size distribution of the cement which controls the packing in the paste and the amount of glass in the clinker. Two cements have received considerable study[5] which seemed to be identical in every respect except for the presence of a much greater hump in the grading curve of one of them. This excess of one size caused interference with the packing of the cement, so that a greater amount of mixing water was needed to fill the voids, leaving a more open structure in the hardened paste. One result was an appreciably greater attack by corrosive solutions.

T. W. Parker of the British Building Research Station[6] has been making a study of the effect of the rate of clinker cooling on the properties of portland cements. Among other things, he observed that those clinkers, cooled sufficiently rapidly to have a high glass content, yielded cements more resistant to sulfate attack than slowly cooled clinkers. He also pointed out that, while most commercial clinkers are comparatively low in glass, a few quite glassy clinkers are produced commercially. Therefore, for proper evaluation of the observed results it might seem necessary to have information on the glass content of the clinkers and on the size distribution of the cements used.

MR. IRA PAUL.[7]—I should like to call Mr. Miller's attention to a laboratory test developed in the New York State Department of Public Works' laboratory which may prove of some interest in determining the sulfate-resistant properties of a portland cement.

About four years ago I started a series of laboratory sulfate-resistant tests on 154 samples of portlant cement which complied with the chemical and physical requirements within the range of the five types of portland cements now promulgated by Committee C-1.[8]

Comparative data were obtained on each sample of cement with the Merriman sulfate test and the Water Test described in a paper presented before the Association of Highway Officials of the North Atlantic States. Briefly stated, the water test on portland cement consists of treating 1 g. of cement with 100 ml. of distilled water and quantitatively determining the percentage of floc liberated at the end of seven days. The details of the test and data are recorded in the 1936 *Proceedings* of the Thirteenth Annual Convention of the Association of Highway Officials of the North Atlantic States, on page 140, and in 1937 *Proceedings* on page 79.

The facts and data which may prove of considerable interest are the correlation between the Merriman sulfate test and the water test. Regardless of the chemical composition of the portland cement, there was a direct relationship between the percentage of floc liberated by the cement in the water test and the number of days at which failure was observed in the Merriman sulfate test. The higher the percentage of floc in the water test the lower the resistance to the sulfate action in the Merriman test. However, the specified 28 day period required in the Merriman sulfate test is insufficient to determine the sulfate-resistant property of the cement. Some failures are likely to occur at 29 days, and up to three months before any reliability can be placed on this test. The claims made on the water test for measuring the sulfate-resistant property of a portland cement are as follows:

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[4] Consulting Specialist on Building Materials, Newark, Ohio.

[5] F. O. Anderegg, "Particle Size Distribution of Portland Cement and What It Means," *Rock Products*, Vol. 43, No. 9, September, 1940, p. 52.

[6] T. W. Parker, "Influence of the Heat Treatment of Portland Cement Clinker on the Properties of Cement," *Journal*, Soc. Chemical Industry, Vol. 58, p. 203 (1939).

[7] Director, Public Works Laboratory, New York State Department of Public Works, Division of Engineering, Albany, N. Y.

[8] Tentative Specifications for Portland Cement (C 150 - 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 167.

1. The water test is a quantitative test method for determining the sulfate resistance of a portland cement.
2. Portland cements liberating floc in excess of 1 per cent are poor sulfate-resistant cements.
3. The sulfate resistance can be determined in 7 days, which is within the period required for other physical and chemical tests.
4. Results of tests are highly reproducible.
5. The technique of the test is very simple, requiring no particular technical skill of operation.

In attempting to tie in the calculated percentage of tricalcium aluminate content of the cement with the water test, it was found that in general the lower the C_3A the less the floc content. However, there were exceptions to this as proven with some occasional cements that calculated to 13 per cent C_3A, which showed no floc in the water test and were highly resistant to sulfate exposure in the Merriman test for a period of over a year. Doubtless, there must have been some differences attributable to the manufacture of such cement to make it sulfate resistant.

It is therefore suggested that the water test be tried out by the authors of the paper presented at this meeting on the cements used in this series of exposure tests, for a possible correlation of their results with our laboratory test.

MR. MILLER.—I was very much interested to hear what Mr. Paul said. We have done very little work with the Merriman test, but I have been told by others who have run that test repeatedly that it did not always correlate with the resistance of the concrete as indicated by exposure tests. Present plans contemplate extending the laboratory tests to include the Merriman and floc tests of these 106 cements, along with other physical tests.

MR. E. C. E. LORD[9] (*by letter*).—The investigations carried out by Ferrari indicate that if the clinker contains sufficient iron to combine with all, or nearly all, the alumina to form C_4AF, a very resistant cement results. Considering the data from this standpoint it will be noted that the average Al_2O_3 of group 1 equals almost exactly that of Fe_2O_3, giving an $\frac{Al_2O_3}{Fe_2O_3}$ ratio of 1.09, this being only 0.45 above the molecular ratio of 0.64 required to form C_4AF. This small excess, reckoned in per cent, would indicate a very small quantity of Al_2O_3 available for the formation of tricalcium aluminate which, if present, would react immediately with the sulfates in the lake water to form calcium sulfoaluminate, the chief cause of cement disintegration. Comparing the $\frac{Al_2O_3}{Fe_2O_3}$ ratio in group 11 it will be noted that the ratio is more than twice that of group 1, indicating a very large percentage of Al_2O_3 to form tricalcium aluminate. This excess alumina might be used as a factor in specifying cement resistance to alkali.

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[9] Burnham Farm, New Castle, Del.; formerly Petrographer, U. S. Bureau of Public Roads; now retired.

MR. A. G. FLEMING[10] (*by letter*).—I would like to congratulate the authors on the excellent work they have been doing on the practical testing of the sulfate resistance of the majority of commercial cements available from the United States and Canada. It is interesting to note that the results of the laboratory tests are confirmed in nearly every case by the Medicine Lake exposure. Since the specimens were buried 4 ft. deep in Medicine Lake they were below the frost line and did not have to endure repeated freezings in addition to the chemical action of the sodium and magnesium sulfates. The photographs of Fig. 1 show that the exposure in 1 per cent magnesium sulfate solutions was more severe in effect than the 1 per cent sodium sulfate solutions, and also the Medicine Lake exposure.

The authors' own comments on the results of their 5-yr. investigation of the sulfate resistance of the 106 cements are brief and to the point. The significant fact to them is that the tests definitely lead to the conclusion that there is a close correlation between sulfate resistance of the cements and the percentage of the calculated compound tricalcium aluminate.

Not one of the cements tested contained 24 per cent of silica, and all of them would therefore have failed to meet the limits for composition proposed for type V cement in the new A.S.T.M. specifications. The maximum silica of the cements under test was 23.6 per cent. Three cements contained that percentage, cement No. 576 placed sixth in group 1, cement No. 614 placed eighth in group 4 and cement No. 594 placed sixth in group 9. The authors rate the cements of group 1 as five or six times as resistant as group 11, and the cement that tops the list contains only 22.5 per cent of silica, while the second in the list contains only 20.4 per cent, and the fifth cement 19.9 per cent. It should appear from these studies that the arbitrary selection of 24 per cent silica as a minimum limit for a sulfate-resistant cement composition is not justified.

Study of the analyses as compared to the performance of the cements under the sulfate exposure tests will show no satisfactory reason for establishing 4 per cent as the upper limit for ferric oxide. Ten of the relatively resistant cements in the two upper groups contained 4 per cent or more. It is advisable perhaps to limit the alumina content to 4 per cent, but it is the ratio of Fe_2O_3 to Al_2O_3 that is important, and from the writer's experience there is little doubt of the advantage gained in better and more uniform burning conditions, especially when silica is being carried high, if the ferric oxide is carried a little higher than 4 per cent. A ratio of Fe_2O_3 to Al_2O_3 of 1.2 or 1.3, and a limit of 4 per cent Al_2O_3 would be easier to meet and will produce a better all-round cement, according to our experience, which as far as the production of sulfate resistant cements is concerned, began in 1930.

The authors in their discussion of their test call attention to the possible need for consideration of the effect of certain minor constituents: magnesia and potassium oxide in particular, and this should be food for thought by other investigators of the problem.

The composition of a cement is undoubtedly important and careful analysis is of great value, but if the limits set for composition are unnecessarily difficult to meet, there is an unnatural hazard set up for good production which may result in cement that will meet the composition limits as determined by chemical analysis but will fall short of the good qualities of a cement slightly easier to manufacture because of the better metallurgical balance. For example, a cement chemist facing a limit of 24 per cent silica in his cement will have to allow for the reducing effect of the coal ash absorbed in the kiln and the gypsum added later to the clinker when it is being ground to cement. He will have some doubt in his mind also regarding the analyses made by the customers' chemist so that to make sure of obtaining enough for the requirement he is likely to make clinker containing about 25 per cent silica. The chemist is also restricted by the 4 per cent limits set for alumina and ferric oxide and in meeting these requirements he is likely to produce a mix deficient in flux content and hence may supply the cement kilns with a refractory mix difficult to burn and hard on kiln linings. Indeed that is the writer's own experience. Under such conditions the resulting product may be of low strength and contain free lime and in general fall short of the desired objective.

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[10] Chief Chemist, Canada Cement Co., Ltd., Montreal, P.Q., Canada.

Moderately high silica content is admittedly desirable, but from our experience the limit set for silica in the proposed type V cement is a little too high and the ferric oxide limit is a little low from a practical operating viewpoint. Is it not advisable to take operating experience into consideration in such matters when seeking to improve a product or certain of its properties? In view of the results obtained by the Minnesota investigation it should be evident that some modification of the composition requirements for type V cement is in order, and it is to be hoped that the members of Committee C-1 will give the matter their immediate attention.

MESSRS. DALTON G. MILLER[3] AND PHILIP W. MANSON[11] (*authors' closure by letter*).—The authors are indebted to Mr. Fleming for bringing into the picture some of the practical problems encountered in the manufacture of a cement to meet specifications that may be theoretically sound but which may be difficult to meet on a quantity basis.

The quantity of silica for the 106 cements ranged from 19.0 to 23.6 per cent and was uniform with respect to quantity and range for each of the eleven groups. The average of Al_2O_3–Fe_2O_3 ratio showed a definite upward trend from 1.09 for group 1 to 2.71 for group 11, with the cements of the more resistant groups showing the greatest ranges.

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[11] Research Instructor, Division of Agricultural Engineering, Department of Agriculture, University of Minnesota, St. Paul, Minn.

THE INFLUENCE OF TEST METHODS ON MOISTURE ABSORPTION AND RESISTANCE OF COARSE AGGREGATE TO FREEZING AND THAWING

By F. N. Wray[1] and H. J. Lichtefeld[1]

Synopsis

Because certain coarse aggregates had been approved on the basis of laboratory tests and later found to be responsible for unsoundness of the concrete in pavements, an investigation studying the relative effects of different testing procedures was undertaken.

Preliminary tests made in 1938 on gravel obtained from its natural source and frozen while in a stream-wet condition showed severe deterioration which could not be duplicated by the usual testing procedure. Because of this disagreement, a definite program of testing was initiated and numerous methods of preparing the aggregate for freezing and thawing were compared.

The relative effects of oven drying and air drying on moisture loss and the increases in the rate and amount of absorption induced by vacuum treatment of the aggregate prior to soaking were investigated. The relation between the degree of moisture saturation and the resistance of the material to freezing and thawing was studied.

One of the outstanding results of the investigation was that the A.S.T.M. tentative method for freezing and thawing produced less deterioration than other methods.

The Missouri Highway Dept. has been investigating test methods for determining the absorption and soundness of coarse aggregates for concrete. Certain coarse aggregates which had been tested according to generally accepted procedures, and based on the test results adjudged to be sound, were later found to be the cause for lack of durability of the concrete in pavements. Casual observation made several years ago and the results of more recent studies of the nature and conditions of concrete failures that were traceable to unsoundness of that aggregate indicated in general the course that the investigation should follow.

The usual procedure employed for years in the Missouri Laboratory for making the freezing-and-thawing tests, was in agreement with the principal provisions of the recently approved A.S.T.M. Tentative Method of Test for Soundness of Aggregates by Freezing and Thawing (C 137 – 38 T)[2] which requires that the aggregate be first oven dried to constant weight and then soaked in water 24 hr. before the start of freezing. The results of these laboratory tests generally showed little absorption and no appreciable deterioration from freezing and thawing even for aggregates which later were adjudged responsible for serious disintegration of the concrete.

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[1] Assistant Research Engineer, and Junior Engineer, respectively, Bureau of Materials, Missouri State Highway Dept., Jefferson City, Mo.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 990.

A few preliminary tests on a particular gravel obtained at its source and taken to the laboratory while in a thoroughly stream-wet condition showed, on oven drying, moisture losses 25 to 50 per cent more than the absorption usually obtained. Like samples of the wet material subjected to freezing and thawing showed astonishing failures at only a few cycles. Samples that were dried, either in the oven or in the air of the laboratory, were found to absorb water very slowly; so in order to accelerate the absorption, some of the dry material was placed in a vacuum for a period of 1 hr. and then inundated with water before normal pressure was restored. This treatment at once proved to be very effective in increasing the absorption of the gravel. Freezing and thawing on this evacuated and reabsorbed aggregate caused deterioration not much less serious than that in the stream-wet material at equivalent numbers of test cycles.

SCOPE

Based on these preliminary facts and indications, an experiment was planned for the study of the following:

1. The effects of different treatments on the amount and rate of moisture loss of a stream-wet or thoroughly saturated aggregate.
2. The effects of different treatments on the amount and rate of moisture absorption.
3. The relationship between deterioration from freezing and thawing and the degree of moisture saturation at the beginning and end of freezing tests.
4. The absorptive properties and resistance to freezing and thawing of different sizes of aggregate particles.

Although the original plan was to make comparative tests on aggregates of different types, no extensive tests including these same methods have as yet been employed on any except one particular source of gravel aggregate. All of the data presented in this report, therefore, pertain to this one material.

The first series of tests, which was started in July, 1938, included eighteen 5200-g. samples, each of which was graded and proportioned as follows:

1300 g. passing a 1½-in. sieve and retained on a 1-in sieve (square opening sieves)
1600 g. passing a 1-in. sieve and retained on a ¾-in sieve (square opening sieves)
2000 g. passing a ¾-in. sieve and retained on a ⅜-in sieve (square opening sieves)
300 g. passing a ⅜-in. sieve and retained on a No. 4 sieve (square opening sieves)

Most of these samples, after being subjected to various processes such as air drying, oven drying, soaking and resaturation following the vacuum treatment, were separated into different size fractions and subjected to repeated freezing in air at 10 F. and thawing in water at room temperature. A little later a supplementary series of samples was added, but each of these consisted of only one size of gravel particles. This series was used only for the study of the relation between particle size and the absorptive characteristics of the material. All of the above-mentioned tests were exploratory in character, and for this reason, the method used for each sample differed from that for every other sample in at least some one phase of treatment.

The last experiment was started in March 1939 and the procedure followed was determined on the bases of definite trends that were observed in the results of the earlier tests. Duplicate samples of about 4500 g. each were used for each treatment and test; and eleven controlled variations in methods were included. The proportions for the different sizes of material in these samples were:

1300 g. passing a 1½-in. sieve and retained on a 1-in. sieve (square opening sieves)
1300 g. passing a 1-in. sieve and retained on a ¾-in. sieve (square opening sieves)
1400 g. passing a ¾-in. sieve and retained on a ⅜-in sieve (square opening sieves)
500 g. passing a ⅜-in. sieve and retained on a No. 4 sieve (square opening sieves)

DISCUSSION OF TEST METHODS AND DATA

Moisture Losses from Stream-Wet Gravel:

Since the preliminary tests had indicated a discrepancy between the amount of moisture originally contained in an aggregate and the 24-hr. normal absorption of the same kind of material, all of the samples in this experiment were prepared from gravel that was stream-wet. All but a few of these samples were dried to constant weight after the initial wet weights were recorded.

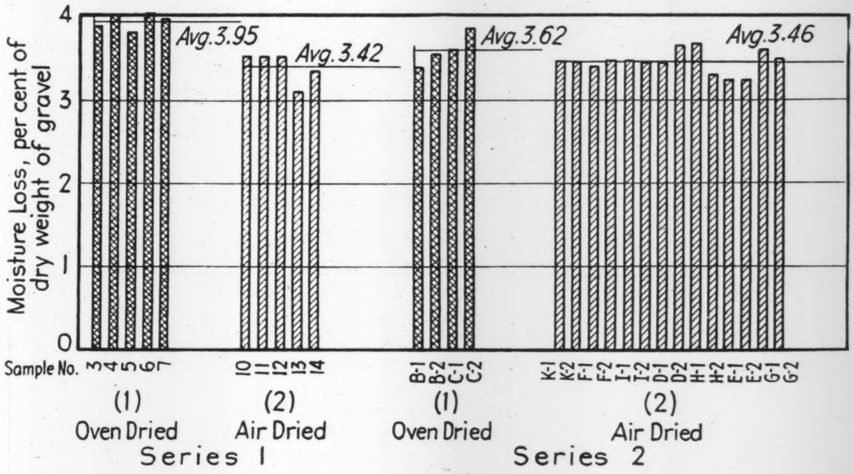

FIG. 1.—Moisture Loss of Stream-Wet Gravel by Drying to Constant Weight.
(1) In oven at 220 F. **(2) In air of laboratory at 75 to 90 F.**

Oven heating at 220 F. of some of the samples expelled appreciably more moisture than that which evaporated from companion samples exposed only to the air of the laboratory. The losses in weight were expressed as percentages of the ultimate dry weights of the respective samples. Figure 1 shows the percentage loss of each sample and the average percentage loss for groups of samples in each series. The tests in series 1 showed a little greater difference between the effect of oven drying and air drying than those of series 2, but this may have been due to slight inaccuracies in the results of Series 1 when less rigid control was maintained.

The rate of air drying and the total amount of moisture lost by each of four companion samples of the stream-wet gravel are shown in Fig. 2. These curves are similar in shape and indicate a required drying time of no less than three weeks for the conditions such as prevailed at that time. The spread of these four curves serves to give an idea of the expected variations in the original moisture capacities of different samples of this kind of material.

Figure 3 shows the influence of particle size on the rate and amount of moisture evaporation from air drying. The finer sizes (curves representing samples 33 and 34) had greater total losses of moisture and, also because of having faster rates of drying, reached conditions of constant weight sooner than the samples of the coarser material. Only the finest size lost all of its moisture by air drying. The three other samples consisting of coarser fractions, and one

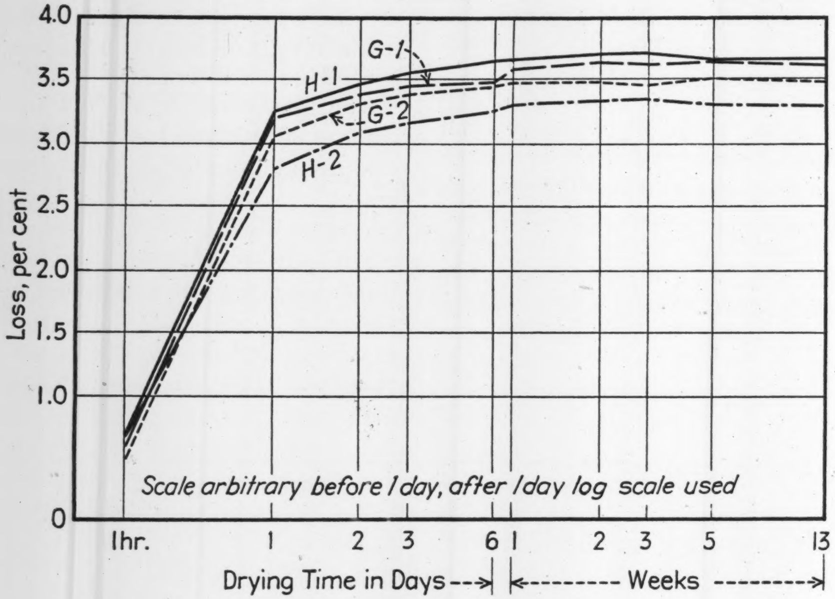

FIG. 2.—Moisture Loss on Air Drying.
Numbers on curves correspond to samples similarly identified in the tests.

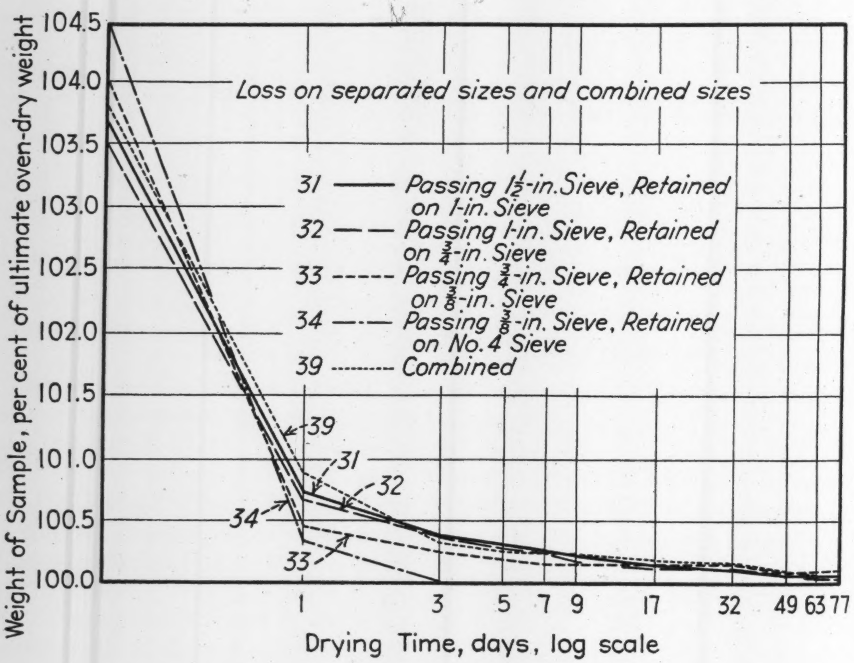

FIG. 3.—Moisture Loss on Air Drying.

sample with four combined sizes, each retained some moisture (about 0.1 per cent) following nine weeks of air drying. This was proved by additional losses when all samples of the group were finally oven dried at 220 F.

Moisture Absorption Tests:

The fact that the amount of moisture lost by the stream-wet gravel was so much greater than the values for absorption found in the records of routine laboratory tests, brings to mind two questions: was it because this material had not the capacity to reabsorb all of the moisture it had once lost; or was it because the usual 24-hr. absorption period was not sufficient? At least partial answers to these questions have now been found.

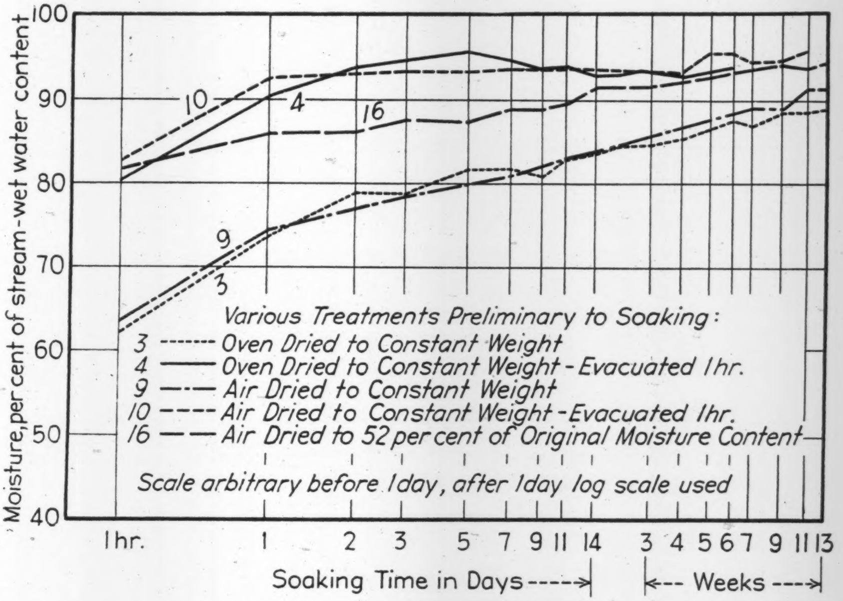

Fig. 4.—Moisture Absorption Tests.

The curves of Fig. 4 show various amounts and rates of moisture absorption in samples that were treated by the several different methods each of which is indicated in the figure. Curves 3 and 9, representing samples of the same numbers, bear out the idea that 24-hr. soaking in water is not sufficient to saturate this type of aggregate. These samples on being soaked 24 hr. took up only about 70 per cent of their total stream-wet moisture content, and then on further soaking, continued to gain in weight throughout a period of 13 weeks without reaching their original degree of saturation. Sample 9 which had been air dried, when compared with sample 3, an oven-dried sample, showed a slightly more uniform rate of absorption and also a little greater total absorption, and there is a possibility that this small difference was due to the effect of oven heating on sample 3.

Curves 4 and 10 (Fig. 4) also represent samples that were oven dried and air dried, respectively, but they, unlike their companion samples, 3 and 9, were placed in a vacuum and maintained at an absolute pressure equivalent to 3 mm. of mercury for 1 hr. prior to wetting.

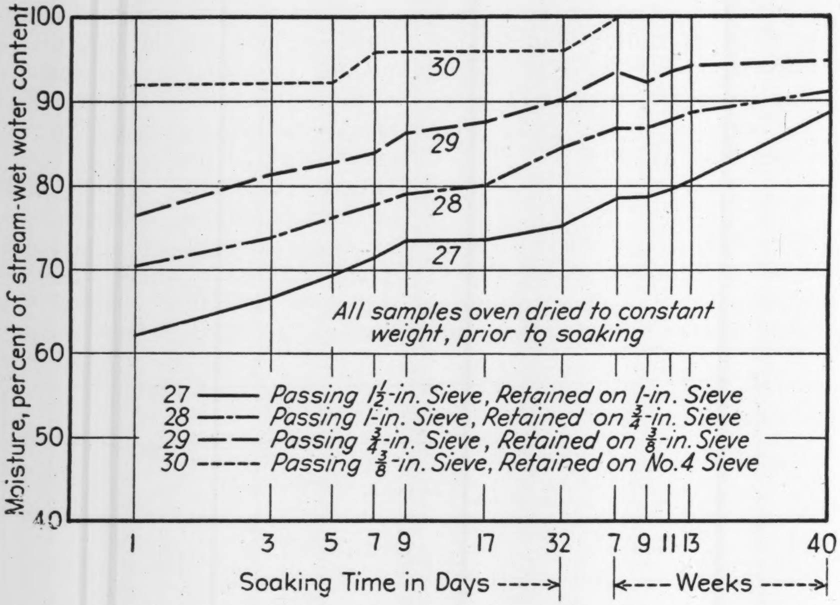

FIG. 5.—Absorption Test on Separated Sizes.

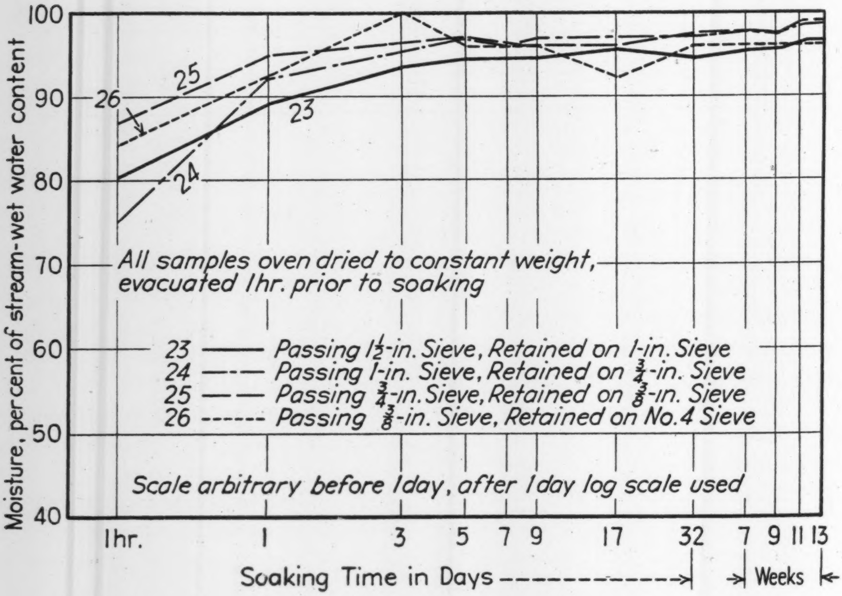

FIG. 6.—Absorption Test on Separated Sizes.

The vacuum was released only as the water was admitted and the gravel was completely inundated before being restored to atmospheric pressure. The absorption proceeded rapidly. At 1 hr. after wetting each of these samples had gained in weight by an amount equal to more than 80 per cent of its stream-wet moisture content. By the end of one day each had gained another 10 per cent, but following that, the rate of absorption became noticeably less. The maximum absorption for these samples appeared to be reached at about 5 to 7 days and this was equivalent to 92 to 94 per cent of original stream-wet capacity. The similarity of curves 4 and 10 for these evacuated samples indicates that the preliminary treatment of oven heating had no significant influence on the reabsorption of water by this material.

Curve 16, in Fig. 4 shows the absorption of a sample of the same material that was only partially air dried before being soaked. Starting from a partially presaturated condition of 50 per cent of its total stream-wet moisture, it gained up to 82 per cent in 1 hr. Thereafter, it had a slow but continuous gain somewhat like that of the other samples which did not receive the vacuum treatment.

Figures 5 and 6 show the relative effects of different sizes of particles on the absorption. A sample consisting of particles of each of four different size groups is represented by a curve in each of these figures. All of the samples were oven dried to constant weight, and then one of the samples for each size was evacuated prior to wetting and soaking, while the other sample of each size group was soaked without the preparatory vacuum treatment. The samples that were not evacuated gave widely different results. The first measurements at the end of 1-day soaking showed the following percentages of moisture regained: 62 for the coarsest fraction, 70 for the next to coarsest, 76 for the next to finest and 92 for the finest size. The three coarser fractions, after the first day, gained about equally for several weeks. At seven weeks the finest size had gained 100 per cent of its original moisture content, but the absorption of each of the other sizes was still considerably less, ranging as low as 78 per cent for the largest particles. The soaking was continued for forty weeks on three of these samples, and only 89 to 95 per cent of their stream-wet water content had been regained.

By comparing the curves of Fig. 6 with those of Fig. 5, it is obvious that the evacuation of the gravel had an extraordinary effect on its absorption. Following 1 hr. in the vacuum and 1 day in water, all samples that had been evacuated were found to be more than 89 per cent saturated, a condition which would have required 40 weeks of soaking without the vacuum treatment. The use of the vacuum proved most effective as an accelerator for the absorption especially for the coarser particles.

Although all of the above data pertain to only one type of gravel aggregate, numerous other tests made in the Missouri Laboratory on various aggregates, including both gravels and crushed stones, confirm these findings qualitatively in so far as the relative effects of different methods of drying and rewetting are concerned.

Freezing-and-Thawing Tests:

The absorption tests which have just been discussed perhaps would not be very significant if it were not for the fact that most of those same samples of aggregate were later subjected to freezing and thawing and that they, in the course

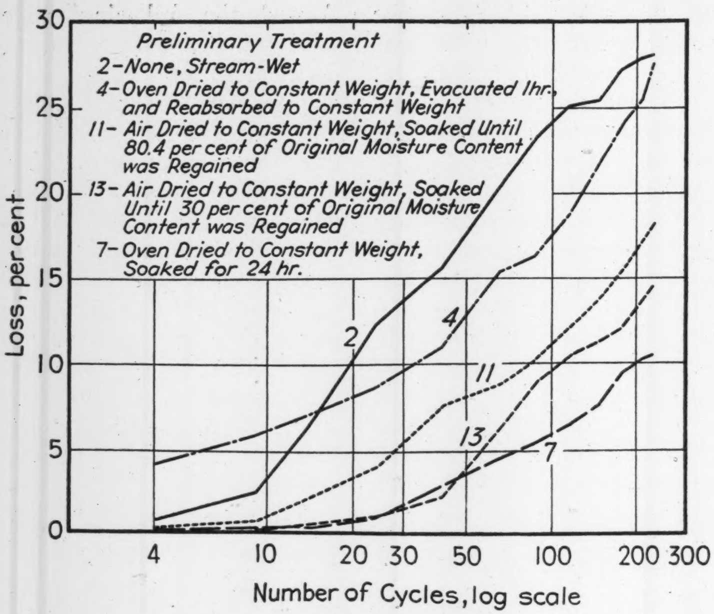

FIG. 7.—Freezing-and-Thawing Test.

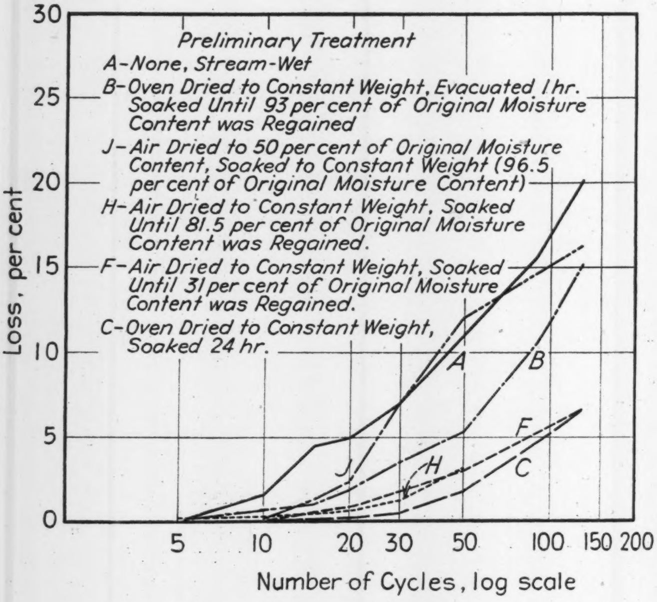

FIG. 8.—Freezing-and-Thawing Test.

of time, exhibited marked influences of the various processes of wetting and drying by which they had been treated.

The procedure for the freezing-and-thawing test in this investigation followed very closely the A.S.T.M. method except for the operations that were designed to vary the moisture content of the aggregate samples. The freezing equipment consisted of a three-compartment heavily insulated box with ammonia coils operated by a York 1-ton, "self-contained" compressor unit. Each compartment of the box had a separate door which opened at the top. As measured inside the coils which surrounded each compartment, the freezing space was 12 in. wide, 36 in. long and 18 in. deep. The temperature was controlled at 10 F. by a thermostat. In arranging the samples for freezing, the various size fractions of the gravel were placed in separate pans, 8 in. square and 2 in. deep. The water was decanted and the pans were set in the box in such a manner as to permit the best possible circulation of the air. The usual freezing cycle consisted of 7 hr. in the freezing chamber and 1 hr. in water at room temperature. Three cycles a day were thus obtained, and this was the procedure followed through most of the experiment. However, occasionally, when it was not possible to change the specimens exactly according to schedule they were retained for longer periods in the refrigerator, rather than in the thawing tank.

Following predetermined numbers of cycles of freezing and thawing each size fraction of every sample was sieved over the same sieve on which it was originally retained. The weight of the material passing this sieve was expressed as a percentage of the weight of the size group before failure occurred. The percentage loss for each whole sample was calculated from the percentage losses of the separated sizes weighted according to definite proportions of sizes determined from representative samples of regular, plant-produced concrete aggregate.

At the times when the samples were being regularly observed the fragments from the failed $1\frac{1}{2}$ to 1-in. particles were sieved over the $\frac{3}{4}$-in. sieve and those from the 1 to $\frac{3}{4}$ in. particles were sieved over the $\frac{3}{8}$-in. sieve, their fractions weighed, and all of the material saved and subjected to further freezing and thawing to show its susceptibility to further deterioration.

In Figs. 7 and 8 are shown the results of the freezing-and-thawing tests on samples which received various preliminary treatments. Figure 7 presents the results obtained on individual samples in series 1 and Fig. 8 the average results obtained on the pairs of duplicate samples in series 2.

Curve 7 in Fig. 7 and curve C in Fig. 8 represent the results obtained on samples treated practically in accordance with the A.S.T.M. tentative method. As is illustrated by the fact that each of these two curves is at the bottom of its group, the samples thus represented had the least deterioration of all the material tested. Other samples which were prepared by methods causing them to contain larger amounts of moisture showed more deterioration. The greatest losses were found in the samples which were stream-wet at the start of freezing; and the next largest losses were in those which had been partially dried and soaked, or evacuated and soaked, to restore the moisture content to about 90 per cent of that which the material contained when stream-wet.

All the curves of Fig. 8 were plotted from the data shown in Table I. This table however presents data for several samples, the curves for which were not plotted. As may be seen in the table, there were no significant variations in the rates or amounts of deterioration of the samples that were air dried to constant weight and soaked without being evacuated. Curve F in Fig. 8 is fairly representative of this entire group of samples which at the start of freezing had degrees of saturation ranging from 4 per cent to 82 per cent. The losses due to failure in these samples were less than 50 per cent as much as those in the stream-wet samples.

The vacuum treated samples, No. 4 of series 1, and B-1, and B-2 of series 2, showed very bad deterioration, but there was no indication that the vacuum treatment had any effect on resistance to freezing and thawing other than the indirect influence of having raised the moisture content of the gravel to a point within the more critical range. These data essentially confirm the findings of Hirschwold and Krueger whose tests have been referred to by Lang.[3]

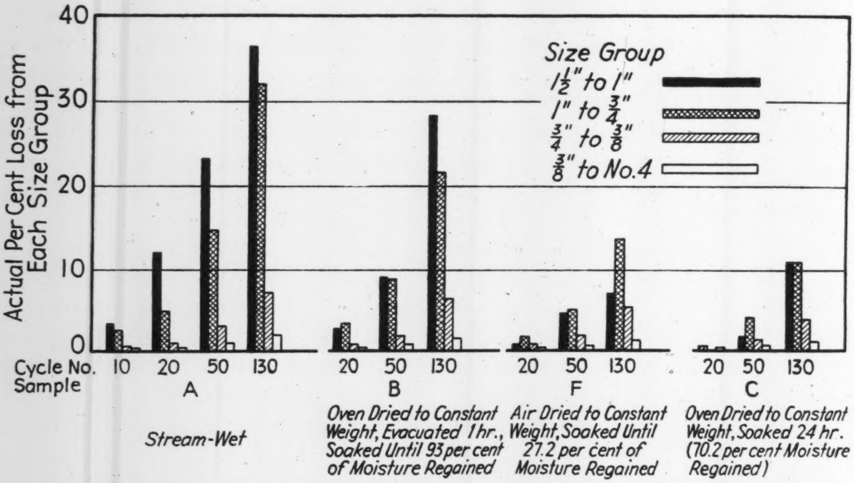

FIG. 9.—Effects of Freezing and Thawing on Different Sizes of Particles.

Curve J in Fig. 8 represents the average results on two samples J-1 and J-2, which had been only partially air dried and then soaked until they were 96 and 97 per cent saturated before being frozen. These were the wettest of all the samples tested excepting those that were 100 per cent stream-wet, and this was the condition most probably responsible for their second highest position with respect to failure in freezing.

Figure 9 shows the loss resulting from freezing and thawing on each size group of particles in each of four different pairs of samples which received different treatments preliminary to freezing. This figure also shows the magnitudes of the losses at different numbers of cycles. Figure 9 illustrates the fact that the larger particles of gravel were responsible for most of the deterioration, and this was typical of the results observed in all of the soundness tests performed under the conditions described.

The apparent influence of particle size on the freezing-and-thawing results was most pronounced in the case of the stream-wet samples and the other samples that were treated and restored to high moisture content before being frozen. The 1½ in. to 1 in. particles exhibited worse failure than the next size, 1 in. to ¾ in., except in the case of the samples which had been dried to constant weight and subjected to freezing without having been raised to a high degree of saturation. The diagrams in Fig. 9, representing samples F and C show instances of the greater losses occurring in the second largest size, 1 in. to ¾ in., and these are some of the exceptions just mentioned. The explanation for this probably lies in the fact that in these particular samples which had been thoroughly dried but only partially reabsorbed, the smaller particles of the two coarse fractions were more nearly saturated at the time of freezing. All of the coarse particles, potentially unsound under the treatment employed, probably had not yet attained a critical moisture content. Substantiating this idea are the curves of Fig. 5, which show that the smaller pieces absorbed considerably more water than the larger ones in the early period of soaking.

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[3] F. C. Lang, "A Discussion on Relation Between Durability of Concrete and Durability of Aggregates," *Proceedings*, Highway Research Board, Vol. 10, p. 118 (1930).

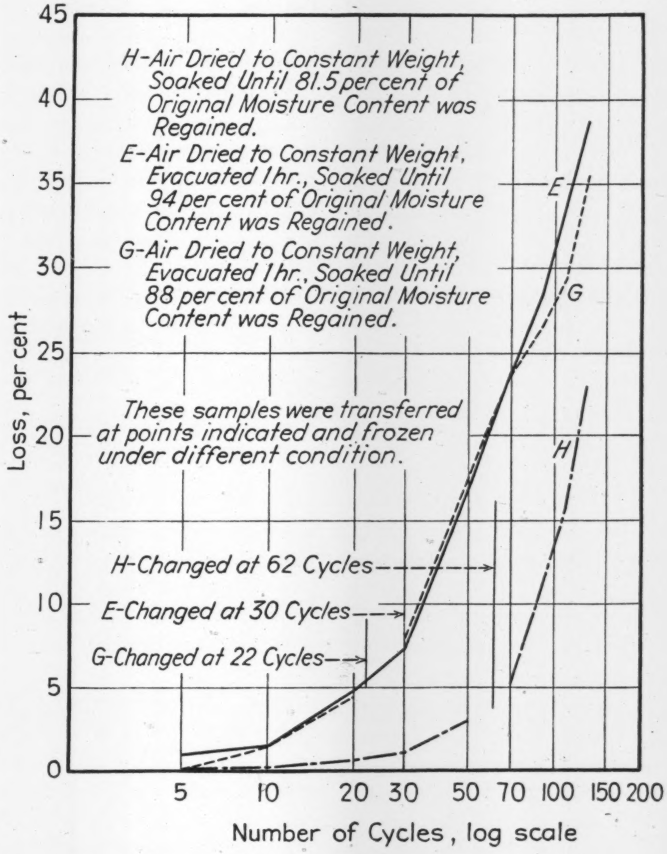

FIG. 10.—Freezing-and-Thawing Tests.

Although some investigators[4] have found worse deterioration on finer sizes, it now appears that one reason for these results may have been that insufficient absorption prior to freezing left the coarser particles with a lower degree of saturation and a correspondingly greater resistance, particularly during the early cycles.

Regarding secondary disintegration or the further failure of fragments originating from failed particles of the larger size groups, the following explanation can be given: In general the same relationships of disintegration, particle size, and degree of moisture saturation found for whole samples appeared also to obtain for the broken pieces. Sieve analyses of the fragments which resulted from failures of particles that were originally retained on the 1-in. sieve showed considerable further breakage of the fragments as the freezing and thawing were continued. The fragments from the failed particles of the $1\frac{1}{2}$ in. to 1 in. group were more susceptible to this secondary breaking than were those from the failed pieces in the next largest size. This was a reflection of the influence of particle size. These second-stage failures were also more extensive among the fragments coming from the stream-wet samples than among those from the other samples, especially the oven-dried material; and this is just a little more evidence that resistance to freezing is affected by the degree of moisture saturation.

The freezing-and-thawing tests on three pairs of samples of series 2 were interrupted due to moving the laboratory. The curves showing the progressive deterioration of these samples are not continuous. They are shown in Fig. 10.

These samples, H, E and G, were the last that were entered in freezing in the old refrigerator and at the completion of 62, 30, and 22 cycles, respectively, the tests were suspended and the aggregates stored in water where they remained for thirty days. The 130 cycles of freezing and thawing originally planned for these samples, were completed at the new laboratory but the results are thought to have been influenced by several changes in conditions none of the effects of which could be evaluated quantitatively. In the later cycles on these samples the freezing temperature was much lower, the rate of freezing was somewhat faster, and the aggregates had rested and soaked in water a month following the completion of several cycles.

Moisture Change During Freezing and Thawing:

At the conclusion of the freezing-and-thawing tests, each sample was oven dried to constant weight and the final percentage moisture calculated and compared with the moisture content which was measured or estimated at the start of freezing.

The values identified as percentage gains in moisture, and shown in the last column of Table I indicate that the stream-wet samples A-1 and A-2 lost a very slight amount of moisture during the 130 cycles of freezing and thawing, and that all of the other samples gained moisture in amounts inversely proportional to their degree of saturation at the start of the tests. Because of the fact that the smallest fragments of the disintegrated material, variable small fractions that passed the No. 4 sieve, were not included in the final moisture determinations, these values for percentage moisture gain may be slightly in error;

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[4] F. C. Lang and C. A. Hughes, "Accelerated Freezing and Thawing as a Quality Test for Concrete Aggregates," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 435 (1931).

yet it is felt that they are sufficiently accurate to show that the freezing and thawing, as employed in these tests did not greatly diminish the moisture of a saturated gravel nor completely saturate one that is less wet.

SUMMARY AND CONCLUSIONS

Great variations in the results of the absorption and the freezing-and-thawing tests were caused by the introduction of several different treatments of the coarse aggregate in its preparation for the tests. The results obtained from these experiments are of value in showing why certain features should or should not be incorporated in any standardized procedure for testing that is intended to furnish information indicative of the behavior of coarse aggregates in service.

1. Thorough drying of the gravel aggregate used in this investigation greatly retarded and diminished the absorption by normal soaking.

2. In this experiment, it was found that a 24-hr. soaking period for the aggregate which previously had been oven dried, was sufficient only to saturate it to about 70 per cent of its original moisture content.

3. Evacuation of samples prior to soaking proved to be a very effective means of rapidly attaining a high degree of saturation.

4. The moisture content of the smaller particles when stream-wet was greater than that of the coarser sizes; also the absorptive capacity and the rate of absorption were greater for the smaller sizes.

5. Stream-wet gravel, which contained the greatest amount of moisture, showed

TABLE I.—RECORD OF FREEZING-AND-THAWING TEST.

Sample Number	Condition or Treatment Prior to Freezing	Moisture at Start of Freezing, per cent	Degree of Saturation, per cent	Loss in Freezing and Thawing, per cent								Moisture Gained, per cent
				5 cycles	10 cycles	15 cycles	20 cycles	30 cycles	50 cycles	90 cycles	130 cycles	
A-1	Stream-wet	3.62	100	0.0	1.4	3.2	4.3	5.9	10.8	14.4	20.2	−0.03
A-2		3.62	100	0.0	2.0	5.7	5.7	8.1	11.3	16.9	20.1	−0.11
Avg.		3.62	100	0.0	1.7	4.5	5.0	7.0	11.0	15.7	20.2	−0.07
B-1	Oven dried, evacuated, soaked to constant weight	3.17	93	0.0	0.5	0.7	1.7	4.3	5.4	11.6	15.8	0.09
B-2		3.27	92	0.0	0.9	1.3	1.8	2.9	5.0	9.5	14.5	0.01
Avg.		3.22	93	0.0	0.7	1.0	1.8	3.6	5.2	10.6	15.2	0.05
C-1	Oven dried, soaked 24 hr. (A.S.T.M.)	2.60	72	0.0	0.0	0.1	0.1	0.4	1.4	4.3	6.6	0.57
C-2		2.66	69	0.0	0.0	0.1	0.1	0.7	2.2	5.1	6.8	0.29
Avg.		2.63	70	0.0	0.0	0.1	0.1	0.5	1.8	4.7	6.7	0.43
K-1	Air dried to constant weight	0.16[b]	4	0.0	0.0	0.0	0.0	0.4	1.7	3.1	4.4	2.79
K-2		0.16[b]	4	0.0	0.0	0.8	1.5	2.7	4.9	7.0	7.8	2.92
Avg.		0.16	4	0.0	0.0	0.4	0.8	1.6	3.3	5.0	6.1	2.85
F-1	Air dried to constant weight soaked	1.13[b]	32	0.0	0.0	0.5	0.7	1.1	2.8	5.0	6.6	1.83
F-2		1.08[b]	30	0.0	0.4	0.4	0.9	2.2	3.2	5.1	6.7	2.02
I-1		2.02[b]	55	0.0	0.2	0.7	1.3	2.7	3.7	6.7	8.4	1.09
I-2		1.88[b]	52	0.2	0.5	0.5	2.3	2.7	3.9	6.0	7.6	1.10
D-1		2.61[b]	73	0.0	0.2	1.3	1.7	2.2	2.3	5.2	6.7	0.47
D-2		2.44[b]	64	0.2	0.2	0.9	1.1	1.4	2.3	4.9	6.1	0.85
H-1[a]		3.10[b]	81	0.1	0.3	0.6	0.9	1.2	3.8	[a]	[a]	[a]
H-2[a]		2.83[b]	82	0.0	0.2	0.2	0.5	1.2	2.4	[a]	[a]	[a]
Avg.				0.1	0.3	0.6	1.2	1.8	3.1	5.5	7.0	
J-1	Air dried 50 per cent soaked to constant weight	3.50[b]	97	0.0	0.4	1.3	1.8	7.4	12.5	15.3	17.7	0.01
J-2		3.47[b]	96	0.0	0.3	1.3	2.8	6.9	11.6	13.9	15.0	0.12
Avg.		3.48	97	0.0	0.4	1.3	2.3	7.2	12.0	14.6	16.3	0.06

[a] These samples transferred and frozen under different conditions in the later cycles.
[b] Estimated percentage of moisture based on oven-dry weight.

the greatest failure on freezing and thawing, while samples prepared according to the A.S.T.M. tentative method which produced only partial saturation showed a minimum of disintegration.

6. The present tentative method for freezing and thawing does not permit, in all cases, thorough saturation prior to freezing and therefore does not provide a positive means of detecting unsound coarse aggregate.

7. The results of these tests definitely indicated that for equivalent degrees of saturation the coarser particles had less resistance to freezing and thawing than the smaller sizes.

8. After freezing and thawing, the fragments resulting from the failure of the original particles contained considerably more moisture than the sound material.

THE SOUNDNESS OF CHERT AS MEASURED BY BULK SPECIFIC GRAVITY AND ABSORPTION

By Charles E. Wuerpel[1] and E. P. Rexford[1]

Synopsis

The object of the studies was to investigate the possibility for isolating the sound and unsound varieties of chert in concrete coarse aggregate by some means more precise than visual examination and more practical than microscopic analysis. Samples of cherty gravel were collected from ten areas in the southern, central and eastern portions of the United States. These were separated into four bulk specific gravity groups by flotation. The separated groups were analyzed microscopically and tested physically for absorptive capacity, resistance to frost action and resistance to a magnesium sulfate soundness test. The test data and discussion of results is preceded by a symposium of data and comments on chert by other investigators. The results of the investigation indicate a close relationship between the void structure (porosity), the bulk specific gravity, and the resistance of most chert particles to freezing and thawing. A procedure is described for mechanically segregating unsound particles of chert from gravel aggregate by flotation.

The tendency for judging the durable quality of *chert* by the notorious bad behavior of some varieties of the rock has resulted in confusion and a generally unfavorable reputation for the classification as a whole.

The word *chert* is used to put a name to an exceedingly wide variety of rocks which have cryptocrystalline or amorphous silica as their groundmass. Chert has been described variously as: any light colored cryptocrystalline rock composed of silica with certain impurities; impure flint; chalcedony, in turn described as cryptocrystalline quartz; a mosaic of quartz and chalcedony, and as amorphous or opaline silica.

Sosman[2] is believed to have given the best "definition" of chert. After describing chalcedony as a "waxy, translucent, natural microcrystalline fibrous silica," and flint "as a cloudy, dark gray, brown or black, opaque natural microcrystalline silica," he includes, as chert, "*any opaque natural microcrystalline or micro-aphanitic silica whose structure and environment do not permit it to be otherwise classified.*"

It seems imperative that the broad term *chert* be subordinated in its connotation of nondurability to a physical procedure for estimating actual relative durability as is done with other general mineral rocks such as granite, sandstone, limestone or "trap." If possible, the unsound varieties of this mineral rock should be grouped with the unsound varieties of other mineral rocks.

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[1] Engineer-in-Charge, and Associate Petrographic Microscopist, respectively, Central Concrete Laboratory, North Atlantic Division, U. S. Engineer Dept., U. S. Military Academy, West Point, N. Y.

[2] R. B. Sosman, "The Properties of Silica," p. 157, Chemical Catalogue Co., Inc., New York, N. Y. (1927).

The purpose of this paper is to present the results of investigations conducted by this laboratory directed toward developing a means for differentiating between sound and unsound chert, together with references to similar investigations conducted by the U. S. Engineer laboratory at Rock Island, Ill. and to available published data which contribute to the fund of knowledge on chert.

TABLE I.—GEOGRAPHICAL LOCATIONS AND IDENTIFICATION SYMBOLS OF CHERTY GRAVELS.

Symbol	Description	Location
S-1	Bank gravel, old alluvial deposit	Northern Mississippi
S-2	Bank gravel, old alluvial deposit	Northern Mississippi
S-3	River gravel	Mississippi River—North of Greenville, Miss.
S-4	River gravel	Mississippi River—South of Greenville, Miss.
NOA	Bank gravel, alluvial deposit	Eastern Louisiana
BC	Glacial deposit	Western New York
W-1	River gravel	Lower reaches—Wabash River
O-1	River gravel	Ohio River—above the mouth of the Wabash
T-1	River gravel	Tennessee River—near its confluence with the Ohio
SW-1	River gravel	Meramec River
SW-2	River gravel	Mississippi River—North of mouth of the Illinois
IRD	Bank gravel	Northwestern Maryland

SAMPLES

The investigation was inspired by the necessity for judging the relative quality and ultimate suitability of gravels in a region in which the total chert content ranged from 50 to 80 per cent of the whole. Samples were collected for analysis from fifteen locations in northern Mississippi. Samples from four of these locations which presented an average representation of the area were chosen for the initial study described herein. The study was extended later to samples of chert-bearing gravels from eight other areas in the United States. The general location from which each of the samples was collected together with the identification symbol of each of the samples are given in Table I.

BASIC RESEARCH

During the early stages of the investigation reference was made to available literature on the subject in an effort to obtain a basic understanding of chert and assistance in segregating the unsound from the sound varieties. The single striking element of agreement present in a majority of the data reviewed was a generalized reference to a connection between the porosity, apparent specific gravity and the durability of chert. In order to minimize the necessity for search for the data referred to, a brief symposium of related comments by other investigators is given below. The gist of the unpublished data collected by the U. S. Engineer Laboratory at Rock Island, Ill. is presented first. Comments which are considered particularly pertinent are italicized by the authors.

TABLE II.—ROCK ISLAND CHERT CLASSIFICATION.

Type	Origin	Description
1.	Gravel deposits	Brown, hard, flinty chert found principally in deposits south of Canton, Mo. Outer surface varying from brown to tan in color. Inside usually lighter shade of brown to tan. Does not break with conchoidal fracture. Generally, dense slightly crystalline structure. Surface usually smooth, but pitted surfaces not uncommon.
2.	Gravel deposits	Solid gray, black, rust or bluish gray chert covered with soft white coating. Group includes stone which has outer surface wholly or partially worn off, having bluish white gravel with grayish mottled center. Center usually crystalline.
3.	Gravel deposits	White colored chert with tiny individual particles of gray and brown throughout. Very hard. Brittle structure. No bedding planes and splinters with a conchoidal break. Surface often pitted. Color sometimes runs in streaks or whorls.
4.	Gravel deposits	Soft chert of dull, white color. Medium coarse in structure. Fractures conchoidally and is slightly fossiliferous.
5.	Stone quarries	Very hard, brittle, white to dirty white chert. Fractures conchoidally and splinters in sharp pieces when broken.
6.	Stone quarries	Chalky, soft, rough textured white chert having porous, amorphous structure, fracturing conchoidally.
7.	Stone quarries	Soft, yellowish or buff chert. Crushes easily with a conchoidal fracture, although the fracture may take another form.
8.	Stone quarries	Chert with thin white or tan coating over a large dark center. Medium hard, fairly dense texture. Crystals may sometimes be present.

TABLE III.—PHYSICAL TESTS OF CHERT (ROCK ISLAND DATA).

Type	$NaSO_4$, 5 cycles, percentage of loss	Heating and Cooling, 30 cycles, percentage of loss	Freezing-and-Thawing Test Free	Apparent Specific Gravity	Absorption, per cent	Silica, per cent	Impurities, per cent[a]
1.	0	None	30 cycles 5%	2.57	0.46	97.4	2.6
2.	6.4	None	30 cycles 14%	2.51	1.72	95.4	4.6
3.	10.4	None	30 cycles 80%	2.48	1.62	97.3	2.7
4.	10.0	None	30 cycles 75%	2.39	4.30	96.8	3.2
5.	100.0	None	20 cycles 100%	2.40	5.10	98.2	1.8
6.	100.0	None	20 cycles 100%	2.12	14.50	98.0	2.0
7.	29.0	None	30 cycles 100%	2.24	10.00	97.0	3.0
8.	3.6	None	30 cycles 75%	2.44	3.71	98.0	2.0

[a] The Rock Island report defines *impurity* as "minerals other than silica."

Symposium:

1. The Rock Island investigation[3] included all available types of chert from gravel pits and quarries along the Mississippi River between Dubuque, Iowa and Reading, Mo. The cherts were separated from the gravels and crushed stones and classified into the eight groups shown in Table II.

The tests performed included "freezing and thawing in free state; sodium sulfate soundness; heating and cooling in free state; freezing and thawing when confined in mortar cubes; heating and cooling when confined in mortar cubes; freezing and thawing until small cracks appeared and then heating and cooling; freezing and thawing of powdered chert confined as one lump in center of mortar cube; chemical composition; freezing and thawing of paraffin-covered chert embedded in mortar cubes; and freezing and thawing of highly absorptive limestone embedded in mortar cubes."

The significant data from the Rock Island tests are shown in Table III.

The results of the freezing and thawing of chert confined in mortar are not given herein, because the number of specimens tested is considered inadequate by the authors and the number of cycles of the test is not listed. However, the report states, "In all cases of failure the embedded chert was predominantly white or buff colored. No crystalline chert caused 'pop-outs'."

The following significant comments on the test results are included in the report.

"Heating and cooling of chert had absolutely no effect. This test was initiated to allay some suppositions that chert caused 'pop-outs' in summertime due to heat of the sun followed by a cooling rain or *vice versa*."

"These results (freezing and thawing of paraffin-coated chert in mortar specimens) indicate that pop-outs occur only from freezing of water absorbed by the chert. This hypothesis is further verified by a comparison of the *absorption* and *specific gravity* of each type of chert with its reaction to freezing and thawing. . . . *It seems a general rule that a chert stone with an absorption greater than 3 per cent or an apparent specific gravity less than 2.50 can be classified as harmful material.*"

"If the specimen (chert pebble) appears amorphous, porous and without life, it can be safely classified as harmful chert. In contrast, if the stone is crystalline, fossiliferous and gives a hard, glossy appearance, it will probably not cause any trouble in concrete."

2. The paper by Spain and Rose[4] makes the following significant statements about unsound chert.

"The lack of soundness may be attributed to several causes. *Chert may be naturally porous*, like much of that from the Knox and Warsaw formations, or *it may reach such a state under long weathering in the river. Because of this porosity, its specific gravity is lowered, and its absorption becomes too high.* Some chert, even though dense and hard, may be so easily fractured and possess so many incipient fracture planes that it will split and disintegrate under freezing and thawing action."

3. Cantrill and Campbell[5] make the following references to the durability of chert.

"Geological definitions of chert vary; however the most widely used defines it as an impure flint and jasper with crystals of pyrite and other foreign material. The color varies from white to gray.... Chert gravel obtained in the western Kentucky area is *extremely porous*, *highly absorptive* and has a *low specific* gravity."

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[3] "A Treatise on Chert," U. S. Engineer Office, Rock Island, Ill., dated May, 1937.

[4] D. L. Spain, Jr., and N. A. Rose, "Geological Study of Gravel Concrete Aggregate," *Technical Publication No. 840*, Am. Inst. Mining and Metallurgical Engrs., October, 1937.

[5] Curtis Cantrill and Louis Campbell, "Selection of Aggregates for Concrete Pavement Based on Service Records," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 937 (1939).

Conclusion 3 of the paper states:

"Chert gravel, which was found to produce unsatisfactory pavement in Kentucky, may be eliminated by two specifications:

(*a*) Coarse aggregate shall not show an *absorption greater than 3 per cent*...."

4. Stanton[6] indicates that the cherty materials, referred to in the California coastal area between Monterey and Los Angeles, had low apparent specific gravity with absorptions ranging from 4.3 to 10.2 per cent. The cherts referred to were principally opaline (amorphous) silica, chalcedonic cherty shale and diatomaceous shale containing opaline silica.

5. Holland[7] states that "The fractured chert which abounds in the Chickamauga area does not ring (when struck), but resounds with a dull thud. The discontinuity of structure which is indicated by this relatively simple test, differentiates between the fractured and the healed cherts. This is also brought out by microscopic tests. The chalky cherts are porous, however, and the absorption and specific gravity tests indicate that it takes considerable time to saturate the pores, but ultimate saturation would result in disintegration by freezing and thawing."

"The fossil chert is ... made up of a mass of easily soluble crinoid stems and other remains embedded in a siliceous cement. On weathering the crinoid stems and other remains were leached away thus allowing the insoluble cement to remain as an extremely porous chert with fossil casts."

"The granular chert is merely a finer textured variety of the fossil chert and would have the same effect in concrete with the exception that it would take a longer time for the results to become evident."

Holland presents also the following tabulation of specific gravities and absorption with comments, as given below:

Description	Apparent Specific Gravity	Absorption, per cent
Gilbertsville coarse, chalky chert.........	1.81	16.7
Gilbertsville fine, chalky chert...............	2.11	6.3
Gilbertsville fossil chert..	1.95	2.5
Chickamauga fractured chert...............	2.49	2.4
Chickamauga massive chert (hard).........	2.65	0.5

"The specific gravity of chert according to Milner varies from 2.59 to 2.64."

"The high absorption and low specific gravity of the coarse chalky and fossil cherts indicate incomplete absorption, hence *discontinuous pore openings*, when compared with the dense chert of specific gravity 2.65 thus showing by computation that about 30 per cent is the actual porosity of the fossil cherts. With the fine chalky chert the tests indicate more complete saturation with a lower percentage of voids as compared with the coarse type...."

"The tests with the fractured chert indicate that almost but not quite complete absorption has taken place. But the tests especially indicate that an otherwise homogeneous material contains extraneous openings of such dimensions that water is able to penetrate them. These openings are of microscopic size and easily enlarged by the freezing of water or extreme temperature changes."

6. F. W. Clarke[8] gives a review of the

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[6] T. E. Stanton, "Expansion of Concrete as Influenced by a Reaction Between the Cement and Aggregate," presented at the meeting of the American Society of Civil Engineers, New York, N. Y., January, 1940 (to be published later).

[7] W. Y. Holland, "Petrography of Aggregates from the T.V.A.," U. S. Department of the Interior, Bureau of Reclamation, Denver, Colo., January 13, 1937.

[8] F. W. Clarke, "Data of Geochemistry," *Bulletin 770*, U. S. Geological Survey (1924).

different theories of the formation of chert, as follows:

"It is at once evident that a considerable variety of rocks may be formed from siliceous oozes, such as the radiolarian and diatomaceous oozes of the Challenger expedition. These fine sediments may be mixed with more or less clay, sand, or calcareous matter, shading when consolidated, into shales, sandstones or siliceous limestones, ... In the same category we must place, ..., such rocks as flint, chert, and novaculite...."

"With some exceptions these rocks are commonly of organic origin...."

"The common variety of compact silica known as chert has been diversely interpreted. A number of writers ... have argued in favor of the replacement theory.... According to F. M. Van Tuyl[a] the cherts of Missouri probably originated in this manner. W. A. Tarr[b], however, and also R. S. Dean[c] argue that the siliceous gel was thrown down from its suspension in sea water by the action of electrolytes and afterward dehydrated and consolidated by pressure."

"On the other hand, chert and flint often exhibit evidences of organic derivation. The radiolarian cherts of California, described by A. C. Lawson, C. Palache, and F. L. Ransome,[d] are principally composed of radiolarian remains. Lawson regards these cherts as having been formed by precipitation of colloidol silica from submarine springs which produced a sort of ooze in which radiolaria became embedded. In other cases cherts were probably derived from sponges, whose spicules consist largely of opaline silica.[e] Cherts crowded with these spicules have been described by various authors, especially W. J. Sollas[f] and G. J. Hinde[g].... The same thing is true of the flint nodules found in chalks, which almost invariably show signs of similar origin. In order to account for these nodules, Sollas suggests that sponge spicules accumulated in a calcareous ooze, where, in presence of sea water under pressure, they partly dissolved. The silica thus taken into solution was later reprecipitated around suitable nuclei, at the same time replacing carbonate of lime. It is possible, however, as A. A. Julian[h] has shown, that the organic matter of decaying sponges may have exerted much influence in bringing about the solution of silica.... There are also siliceous concretion-like flints, as well as oolites which are formed by the deposition of silica from solution around quartz grains .... It is possible that a single formation may represent more than one of these processes. R. D. Irving and C. R. Van Hise[i]... describing the chert of the Penokee iron region ... suggest that it may have been partly derived from organic remains and also be partly a chemical sediment. In short, no one process can account for all the occurrences of amorphous or cryptocrystalline silica, and each locality must be studied in the light of its own evidence."

7. The following comments on the formation of chert are taken from Twenhofel:[9]

"Chert and kindred rocks are among the most common in the geologic column. All are composed of silica and various impurities. Textures are fine grained to cryptocrystalline, and *to the unaided eye seem amorphous.* Chert includes varieties that have white, gray, and other light colors, and flint those of dark gray to black colors. . . ."

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[a] *American Journal of Science*, Fourth Series, Vol. 45, p. 449 (1918).

[b] *Idem*, Vol. 44, p. 409 (1917).

[c] *Idem*, Vol. 45, p. 411 (1918).

[d] Lawson, *Fifteenth Annual Report*, U. S. Geological Survey, p. 420 (1895).

Lawson and Palache, *Bulletin*, Department of Geology, University of California, Vol. 2, pp. 354, 365 (1902).

Ransome, *Idem*, Vol. 1, p. 193 (1894).

[e] Thoulet, *Bulletin*, Soc. min., Vol. 7, p. 147 (1884).

[f] Ann. and Mag. Nat. Hist., Fifth Series, Vol. 6, pp. 384, 437 (1880); Vol. 7, p. 141 (1881).

[g] *Philosophical Transactions*, Vol. 176, p. 403 (1885); *Geological Magazine*, p. 435 (1887); *Idem*, p. 241 (1888).

[h] *Proceedings*, Am. Assn. Advancement Science, p. 396 (1879).

[i] *Tenth Annual Report*, U. S. Geological Survey, Part 1, p. 397 (1890).

See also C. R. Van Hise, "A Treatise on Metamorphism," Mon. U. S. Geological Survey, Vol. 47, pp. 847-853 (1904).

[9] W. H. Twenhofel, "Treatise on Sedimentation," McGraw-Hill Book Co., Inc., New York, N. Y. (1939).

"Common impurities are hematite, limonite, pyrite, marcasite, carbonaceous matter, and calcium and magnesium carbonate."

"The silica in the Mesozoic and older cherts and flints is crystallized and in thin section shows a mosaic of quartz and chalcedony. . . . Some of the younger cherts and flints contain opaline or amorphous silica. Weathered chert becomes white and chalklike."

"Some cherts and flints contain considerable calcium carbonate, and some grade laterally into limestones. . . ."

"The color of some cherts is uniform throughout, in others the colors are arranged in irregular patches so as to give a mottled appearance, in still others the colors are arranged in bands. . . ."

"The colors are usually due to some impurity, but they may be due to differences of texture or porosity. . . ."

"Nodulus cherts and flints are usually associated with limestones. Flints are mostly in chalk and cherts in other varieties of limestones. Bedded cherts and flints have association with all kinds of rocks. . . ."

"Chert and flint may serve as cement for other sediments, but occurrences are of limited extent. . . ."

8. G. P. Merril[10] makes the following significant comments on chert:

"Weathering of chert.—Among siliceous sedimentary rocks poor in alkalies or iron-bearing silicates the degeneration is mainly disintegration, though a small amount of silica, existing in either crystalline or chalcedonic forms, is usually lost through solution. Thus the cherts of southwest Missouri break down into porous friable forms, sometimes passing into the condition of loose powder. . . ."

"Analyses of fresh and altered forms of this material, as given by Dr. E. O. Hovey,[a] show no differences that are of sufficient importance to warrant us in assuming any of them as direct causes of disintegration. The change is mainly physical, though it is more than probable that a certain amount of interstitial silica has been removed. . . . Dr. Penrose, however, describes[b] a process of chert decay, or more properly disintegration, as manifested in the Batesville region of Arkansas, in which the cause of the breaking down is more apparent. There are two stages in the process, as described: (1) A transition into a light, porous, opaque, buff-colored rock of the consistency of ordinary pressed brick, and (2) into an impalpable white or brown powder, locally known as a polishing powder. . . . The white residual powder often contains masses of the porous, semidecomposed rock, the latter in turn encircling kernels of hard, unaltered chert. Throughout this region, the cherts (of Carboniferous age) are generally decomposed into the condition of a more or less porous mass. . . . In all cases the disintegration may be traced to the removal, by leaching, of a small amount of interstitial carbonate of lime."

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[a] Appendix A, Missouri Geological Survey, Vol. VII, pp. 727-739 (1894).

[b] *Annual Report*, Geological Survey of Arkansas, Vol. 1 (1890).

PHYSICAL TESTS

Appraisal of the gravel particles in the four basic samples of the investigation (listed as S-1 to S-4 in Table I), in terms of noncherts and cherts of varying weathering characteristics, was attempted by (1) visual examination using the classification of chert suggested in the Rock Island[3] report, (2) by microscopic examination, (3) by freezing and thawing in unconfined and confined (in concrete) conditions, (4) by the magnesium sulfate accelerated soundness test,[11] (5) by absorption, and (6) in terms of bulk specific gravity separation in a flotation test. These tests were followed by a detailed investigation of the eight additional samples listed in Table I.

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[10] G. P. Merril, "Rock, Rock Weathering and Soils," MacMillan Co., New York, N. Y. (1906).

The terms "absorption" and "bulk specific gravity" referred to herein are based upon a 5-hr. period of boiling in water instead of the 24-hr. immersion period requirement of A.S.T.M. Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 39).[12] The formulas used in determining the factors are as follows:

TABLE IV.—RELATION BETWEEN BULK SPECIFIC GRAVITY, ABSORPTION AND SOUNDNESS OF CHERT BASED UPON VISUAL CLASSIFICATION OF FIVE TYPES IN THE "S" GRAVELS.

Type	Bulk Specific Gravity					Absorption, per cent					Soundness Magnesium Sulfate									
											5 cycles, percentage loss in weight					10 cycles, percentage loss in weight				
	S-1	S-2	S-3	S-4	Avg.	S-1	S-2	S-3	S-4	Avg.	S-1	S-2	S-3	S-4	Avg.	S-1	S-2	S-3	S-4	Avg.
Nonchert	2.6	2.6	2.6	2.6	2.6	1.6	1.8	1.7	1.7	1.7	2.0	1.3	0.5	1.7	1.4	3.6	2.9	1.4	1.8	2.4
I	2.6	2.6	2.6	2.6	2.6	1.8	1.3	0.8	0.6	1.1	0.1	0.1	0.0	0.2	0.1	0.2	0.1	0.0	0.2	0.1
II	2.5	2.5	2.6	2.5	2.5	3.4	2.7	1.7	2.1	2.5	1.2	1.5	0.8	0.0	0.9	2.1	2.0	0.2	0.0	1.1
III	2.5	2.6	2.6	2.6	2.6	3.2	1.3	1.2	1.6	1.8	2.6	1.5	1.4	6.8	3.1	4.9	1.6	1.6	6.8	3.8
IV	2.5	2.5	2.5	2.5	2.5	5.0	3.6	3.7	3.7	4.0	5.0	2.1	2.3	3.2	3.2	6.3	3.5	3.1	3.7	4.1
V	2.2	2.4	2.4	2.3	2.3	11.9	7.4	7.2	9.5	9.0	36.6	16.4	13.2	9.0	19.0	45.8	19.4	14.9	17.5	24.4

$$\text{Absorption} = \frac{B - A}{A}\,100$$

Where B is the saturated surface dry weight after 5 hr. of boiling in water and A is the dry weight.

$$\text{Bulk specific gravity} = \frac{B}{B - C}$$

Where C is the weight of the boiled sample immersed in water.

True specific gravity is the ratio of the dry weight to the displacement of the powdered material. Displacement was measured in a Le Chatelier flask.

Visual Examination:

Typical specimens consisting of about 500 pebbles ($\frac{3}{8}$ to 1 in. in size) of each of the "S" samples were examined under a 10× lens in an attempt to segregate the chert particles into the eight Rock Island classifications. Each of the approximately 2000 pebbles were broken by hammer blows, before being examined under the lens, so as to expose unstained cross-sectional areas. The separated portions of each of the eight classifications were tested for bulk specific gravity, absorption and soundness in the magnesium sulfate test. The results were irregular and contradictory. The procedure was repeated using five instead of eight chert classifications and the concordance between specific gravity, absorption and soundness was much improved and generally significant. The results of these tests are given in Table IV.

A description of each of the five types of chert is not given, because such descriptions are felt to lead to false values and because the length of time required to make the five classifications and the tedious examination of each broken particle under a lens detracts materially from the value of this method as a practical analysis.

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[11] C. E. Wuerpel, "Proposed Method of Test for Soundness of Aggregates by Use of Magnesium Sulfate," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 885 (1939).

[12] 1939 Book of A.S.T.M. Standards, Part II, p. 300.

Flotation Separation:

In view of the evidence of the relationship between bulk specific gravity and the weathering quality of chert, it appeared that a flotation test might be serviceable as a means of mechanical separation of chert types. The flotation test procedure described below was devised to test this theory.

(*a*) *Solution.*—Solutions of bromoform (sp. gr. 2.86) and mono-bromobenzene (sp. gr. 1.46) were prepared in 500 ml., quantities having specific gravities of 2.30, 2.40 and 2.50. The solutions were maintained at the exact gravity desired by use of a special hydrometer having a range of 2.00 to 3.00 specific gravity marked in increments of 0.01 specific gravity and accurate to 0.02 specific gravity throughout its range. The flotation solutions are slightly volatile and require readjustment to the desired gravity by the occasional addition of mono-bromobenzene. The mono-bromo-benzene is inflammable, but no danger or fire-hazard will exist if flame does not come into contact with the liquid.

TABLE V.—RELATION BETWEEN BULK SPECIFIC GRAVITY, ABSORPTION AND SOUNDNESS OF CHERT BASED UPON FLOTATION SEPARATION OF "S" GRAVELS.

Bulk Specific Gravity	Absorption, per cent				Soundness Magnesium Sulfate							
					5 cycles, percentage loss in weight				10 cycles, percentage loss in weight			
	S-1	S-2	S-3	S-4	S-1	S-2	S-3	S-4	S-1	S-2	S-3	S-4
+2.50	1.5	1.0	1.0	1.0	0.7	0.8	0.0	0.1	2.1	1.9	1.7	1.8
2.40 to 2.50	2.6	2.2	2.4	2.5	2.9	1.3	1.1	1.0	5.4	3.5	4.3	3.1
2.30 to 2.40	6.1	4.2	4.4	4.3	7.3	4.2	3.6	3.4	11.4	6.7	9.3	6.2
−2.30	13.9	8.0	7.2	7.6	9.3	12.9	5.9	4.6	36.0	22.4	17.3	13.4

(*b*) *Solution Container.*—Heavy glass conical graduates or large separatory funnels were used as solution containers during the flotation procedure. The conical graduate is peculiarly well adapted to the procedure, because it provides adequate depth to float the hydrometer in 400 ml. of solution with sufficient surface diameter to manipulate the pebbles and pebble spoon. When not in use the solutions were kept in tightly stoppered bottles.

(*c*) *Test Sample.*—The test sample consisted of 10 lb. (approximately 470 pebbles) of gravel ranging in size from $\frac{3}{8}$ in. to 1 in., in diameter. In cases where the gravel investigated had a maximum pebble size of 2 in. a parallel sample 20 lb. in weight ranging from 1 in. to 2 in. in size was included in the procedure.

(*d*) *Sample Preparation.*—The sample described in (*c*) was placed in water and boiled for 5 hr., cooled, hand-dried to a saturated surface-dry condition and then weighed. After weighing, the sample was covered with a damp cloth to prevent further drying prior to immersion in the flotation solution.

(*e*) *Flotation.*—The saturated surface-dry pebbles were introduced into the 2.50 specific gravity solution in increments of about 20 pebbles at a time. The pebbles which floated, having a gravity less than 2.50, were removed and introduced into the 2.40 solution. The pebbles which sank in the 2.50 solution were removed and placed in water in a container marked +2.50. The process of separation was continued by placing the "floaters" in each solution into the solution of next lower gravity. The finally separated pebbles were then in groups identified as +2.50, 2.40 to 2.50, 2.30 to 2.40 and −2.30. The bromoform solution, being immiscible with and heavier than water, separated from the surfaces of the pebbles and settled to the bottom of the containers in which the different fractions were placed.

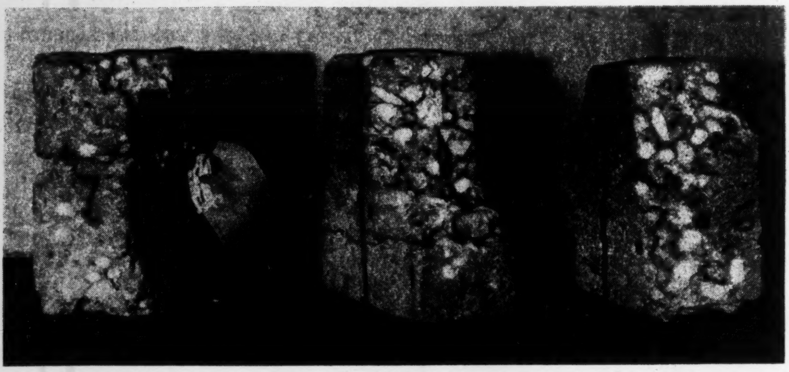

FIG. 1.—Concrete Slabs Split by Pebbles of − 2.40 Chert Which Ruptured in a Freezing-and-Thawing Test.

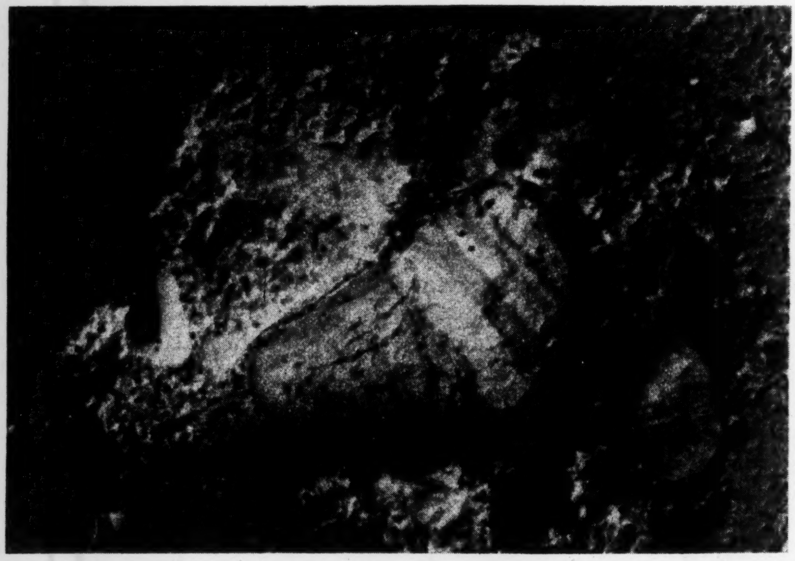

FIG. 2.—Typical Surface Pop-out, Showing Highly Porous Chert Particle with a Specific Gravity Less Than 2.40.

The separated portions of the four "S" gravel samples were subjected to tests for absorption, soundness (magnesium sulfate test) and freezing and thawing. The increase in absorption and decrease in soundness with lower bulk specific gravity of these samples are shown in Table V.

It will be noted from the data in Table

V that there is a marked increase in absorption and a marked decrease in soundness in the pebbles having a bulk specific gravity of less than 2.40.

The results of the freezing-and-thawing tests of the separated samples confined in concrete disks (4 in. deep and 6 in. in diameter) and slabs (8 by 10 by 3 in.) corroborated the indications of soundness by showing diminished resistance to freezing with decrease in bulk specific gravity. However, the results were not reducible to quantitative tabulation. In some cases, the test specimen split into two or more sections; in other cases the failure was by surface "pop-outs" and in others by general disintegration. A typical example of the splitting action is shown in Fig. 1 and a typical "pop-out" is shown in Fig. 2. However, it was definitely observed that the disrupted pebbles were all of the groups lower in specific gravity than 2.50 and the relative percentage of failures increased markedly with decrease in pebble gravity in all of the "S" gravels.

Field Observations:

The results of the laboratory tests on the "S" gravels were checked by a survey of the exposed concrete structures in the area from which the gravels had been obtained. A group of 100 of the roughly conical sections having a portion of disrupted chert at the apex, known as "pop-outs," were collected from representative structures. The chert particles were removed from the pop-outs and tested for absorption and bulk specific gravity. In every case the particle had an absorption greater than 4 per cent and a bulk gravity less than 2.40.

TABLE VI.—RELATION BETWEEN BULK SPECIFIC GRAVITY, ABSORPTION AND SOUNDNESS OF CHERT GRAVELS.

Sample	Bulk Specific Gravity, +2.50			Bulk Specific Gravity, 2.40 to 2.50			Bulk Specific Gravity, 2.30 to 2.40			Bulk Specific Gravity, −2.30		
	Distribution, per cent	Absorption, per cent	Loss, per cent[a]	Distribution, per cent	Absorption, per cent	Loss, per cent[a]	Distribution, per cent	Absorption, per cent	Loss, per cent[a]	Distribution, per cent	Absorption, per cent	Loss, per cent[a]
S-1	36	1.5	2.1	19	2.6	5.4	15	6.1	11.4	29	13.9	36.0
S-2	51	1.0	1.9	32	2.2	3.5	10	4.2	6.7	7	8.0	22.4
S-3	55	1.0	1.7	29	2.4	4.3	12	4.4	9.3	4	7.2	17.3
S-4	54	1.0	1.8	27	2.5	3.1	13	4.3	6.2	6	7.6	13.4
NOA	81	0.3	2.5	12	2.3	4.0	6	4.5	11.0	1	9.1	18.0
BC	91	0.1	10.2[c]	7	1.9	2.0	2	5.6	5.0	0	....	....
W-1	86	1.3	2.6	8	3.7	5.8	4	5.8	13.1	2	9.4	45.7
O-1	79	0.4	2.6	12	2.7	6.0	6	4.7	9.0	3	9.1	20.4
T-1	14	2.1	7.3	37	3.3	4.5	30	5.6	11.1	19	7.3	14.2
SW-1	67	1.5	3.1	28	3.6	7.2	2	5.8	[f]	3	9.7	[f]
SW-2	96	2.5	2.3	0	..	[f]	1	4.6	[f]	3	9.5	[f]
IRD	[b]	1.2	4.5	95[e]	1.6	7.4	5[g]	7.8[g]	20.1[g]	[b]	[b]	[b]
Average	..	1.1	3.0[d]	..	2.6	4.8	..	5.4	10.3	..	9.1	23.4

[a] Loss in weight after 10 cycles of the magnesium sulfate soundness test.
[b] Not determined.
[c] High loss due to presence of unsound sandstone.
[d] Excluding extraneous BC value.
[e] Range from 2.40 up to maximum present.
[f] Insufficient sample for test.
[g] Range from 2.40 down to minimum present.

EXTENDED PROGRAM

The unusually excellent relationship which was found to exist between the absorption, bulk specific gravity and soundness of the "S" gravels suggested the desirability for verifying the relationship by similar tests of cherty gravels from other localities.

Samples were collected from the eight additional regions shown in Table I, by persons not connected with this laboratory. The specific origin or the degree of representation of the gravels is not known. However, they are all glacial or alluvial gravels which were known to contain chert.

The results of the flotation separation and subsequent tests for absorption and soundness in magnesium sulfate of these additional samples are given in Table VI. The relation between absorption, bulk specific gravity and soundness are illustrated best by the average values shown at the bottom of the table. Again, the increase in absorption and decrease in soundness is most marked as the bulk specific gravity drops below 2.40.

The freezing-and-thawing tests made on these gravels contained in concrete were in accord with the results observed from the S gravel tests, with the exception that the concrete specimens containing the first samples of the T-1 gravel in the +2.50 group failed after 15 cycles. Inspection of the particles of gravel causing the failures disclosed a shattering of certain particles along definite planes of cleavage. Immediate microscopic examination of some of the particles which had shattered showed that the particles contained an unusually high iron content. The concrete freezing tests were repeated with the T-1, +2.50 gravel and at the time of writing (50 cycles) no distress of the samples of any sort has been observed. However, the possible presence of chert particles with planes of weakness formed by cracks filled with iron compounds of high gravity was recognized by the failure of the initial T-1, +2.50 specimens. The microscopic investigation detailed below indicates the probability that such particles may exist in other cherty gravels.

MICROSCOPIC INVESTIGATION

All of the cherts encountered in this investigation were of the cryptocrystalline variety and were composed predominantly of quartz. Even in cases of extremely fine-textured cryptocrystalline structure, high magnification showed the optical properties of the minute individual crystals to be those of quartz. When distinctly recognizable chalcedony occurred it was in the replacement of fossils or in the filling of seams. The "mosaic of chalcedony" as reported by Holland[7] was not observed. The only case in which opaline silica was definitely found was in samples of chert received from California.[13] The samples were representative of a localized area in California and were requested for comparative purposes.

While it is not intended to imply that opaline silica may never occur in intimate association with a cryptocrystalline structure, it is intended to emphasize the fact that it did not occur in any of the samples of cherty gravel included in this investigation, in sufficient quantity to be identified and therefore to be of practical significance.

Thin sections were prepared from seventy typical pebbles and examined by means of the petrographic microscope. A number of polished sections also were studied microscopically, by reflected light. Innumerable chert pebbles from all the localities were examined macroscopically and microscopically in powder sections.

Variable amounts of hematite (Fe_2O_3), limonite ($Fe_2O_3 \cdot x\ H_2O$), calcite ($CaCO_3$) and carbonaceous matter occurred as the principal impurities. The amounts of the different impurities varied widely from a bare trace to a considerable proportion of the pebble.

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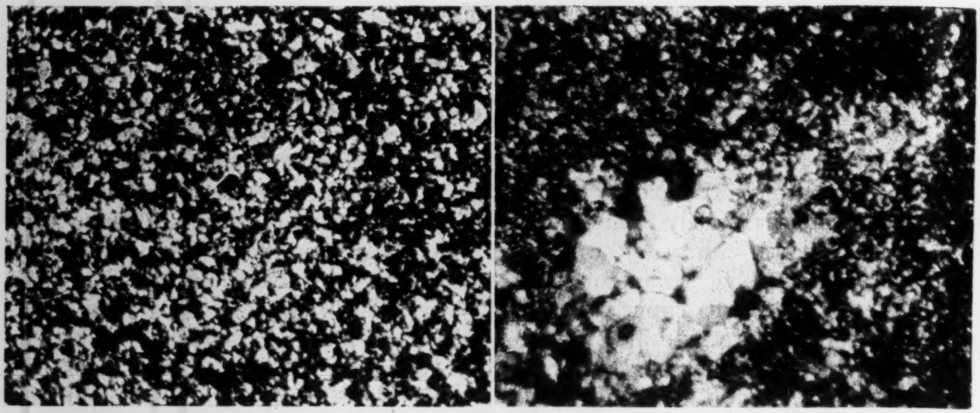
[13] Provided by T. E. Stanton, California Division of Highways.

The structure and texture of the pebbles also varied widely from a dense, hard, glassy type with a uniform compact cryptocrystalline groundmass, to the comparatively soft porous type with a nonuniform cryptocrystalline groundmass enclosing an abundance of voids and pores of variable size and shape. In some cases, particularly in the dark brown colored cherts, voids and cracks were filled with an oxide or hydroxide of iron, some clay and organic matter. In other cases they were sealed with a secondary development of silica in the form of quartz or chalcedony.

The arrangement of the thin sections from each locality in order of bulk specific gravity of the original pebbles indicated that there was usually a close relationship between bulk specific gravity, pore structure, and durability. Pebbles having a bulk specific gravity greater than 2.50 were, in the majority of cases, compact, dense cryptocrystalline masses composed largely of quartz with minor, but variable, amounts of chalcedony, iron oxide, iron hydroxide, calcium carbonate and carbonaceous matter (Fig. 3(*a*), (*b*), and (*c*)). Open voids and cracks were generally absent and when they did occur they were not significant features of the structure. Healed cracks or seams were sometimes present, but they were sealed by a secondary development of silica or a form of amorphous iron oxide. Pebbles having a bulk specific gravity of less than 2.30, in contrast to the +2.50, were characterized generally by numerous open voids of variable size, but often sealed from each other by the dense cryptocrystalline groundmass (Fig. 3(*d*), (*e*), and (*f*)). These pebbles, like the ones having a bulk specific gravity of +2.50, were cryptocrystalline masses composed largely of quartz with variable amounts of chalcedony, iron hydroxide, iron oxide and carbonaceous matter. In fact the only difference evident between the +2.50 fraction and the −2.30 from any one locality was the abundance of voids in the latter fraction.

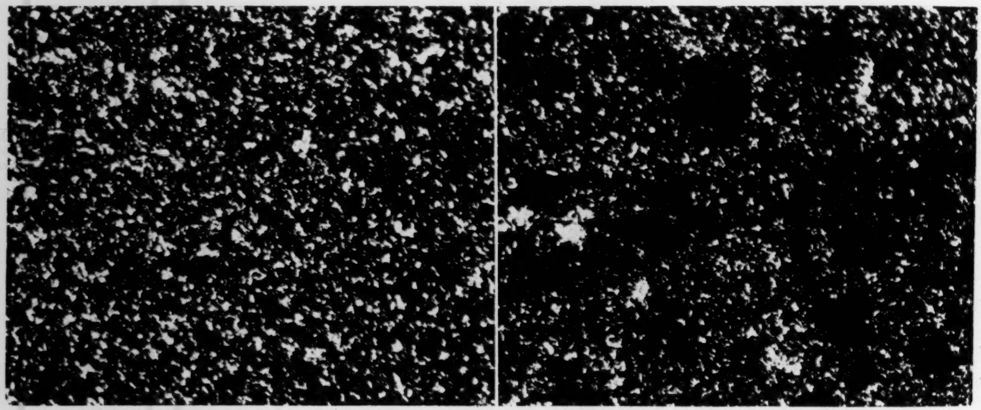
A further substantiation of the basic similarity in mineral composition of the +2.50 and −2.30 cherts, from the same locality, was found by determining that their true specific gravities were within 0.02 of the specific gravity of quartz (2.65), although their bulk specific gravities varied from 2.10 to 2.65.

While it is not intended to overemphasize a condition which occurred to any material extent in pebbles from only one of the localities studied (T-1), it is considered that some discussion is desirable concerning those pebbles having a bulk specific gravity of +2.50 which contained numerous cracks, seams and voids "sealed" by amorphous iron oxide. Such pebbles are sometimes deceiving in that although they may have a bulk specific gravity of +2.50 and appear to have a hard, dense structure, they have been found to fail in a concrete freezing-and-thawing test. These pebbles are usually of a darker brown color than their more satisfactory neighbors and frequently have a rather dull luster. Under the microscope, they show no open voids or cracks, but do show an abundance of small areas or seams of amorphous iron oxide intimately associated with the cryptocrystalline silica groundmass (Fig. 4(*a*), (*b*), and (*c*)). When these pebbles are placed in hot Canada balsam or evacuated in liquid bakelite, in preparation for thin sections, it will be many minutes before the coating of minute air bubbles which cover the specimen disappear. This indicates the presence of innumerable submicroscopic pores which



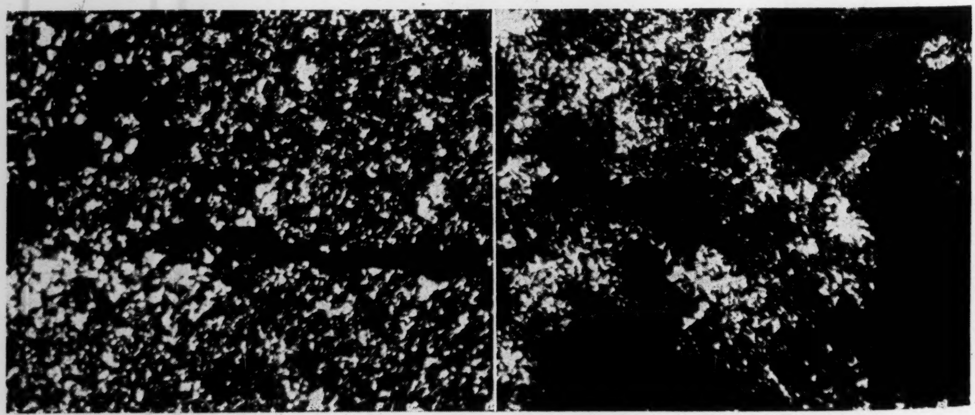
(*a*) +2.40 chert of class 1. Shows coarse textured cryptocrystalline silica in different degrees of optical orientation. No voids present.

(*b*) +2.40 chert of class 1. Shows large quartz crystals in a cryptocrystalline silica groundmass. No voids present.



(*c*) +2.40 chert of class 2. Shows fine textured cryptocrystalline silica in different degrees of optical orientation. No voids present.

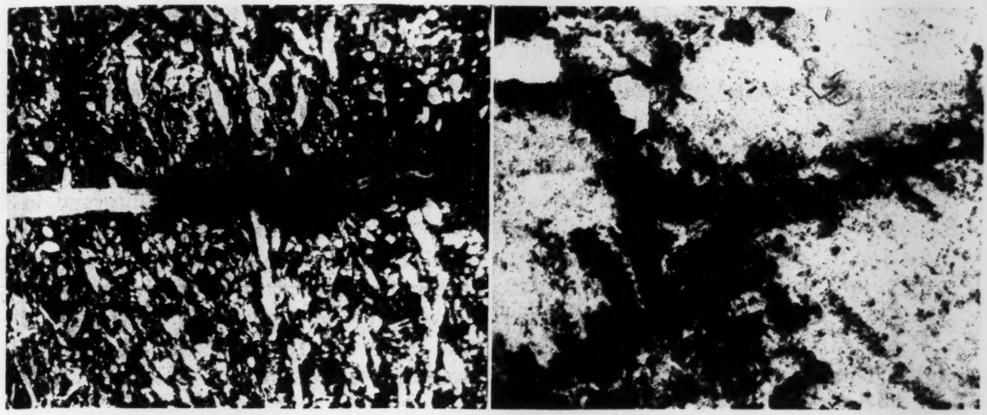
(*d*) −2.40 chert of class 2. Shows voids (larger round black areas) sealed from each other by the cryptocrystalline silica groundmass.



(*e*) −2.40 chert of class 2. The rounded black areas and the black streak are open voids and an *internal* crack respectively.

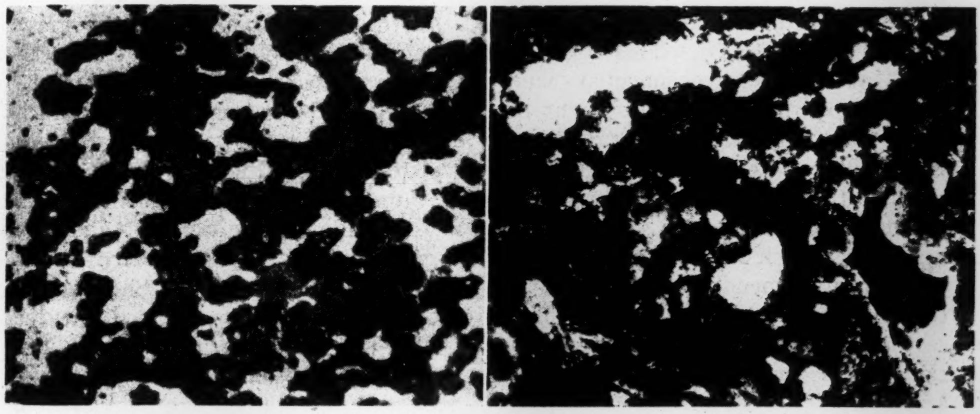
(*f*) −2.40 chert of class 1. Shows large voids (black) in a relatively coarse cryptocrystalline groundmass.

FIG. 3.—Thin Sections of Chert in Gravel, Crossed Nicols (× 100).



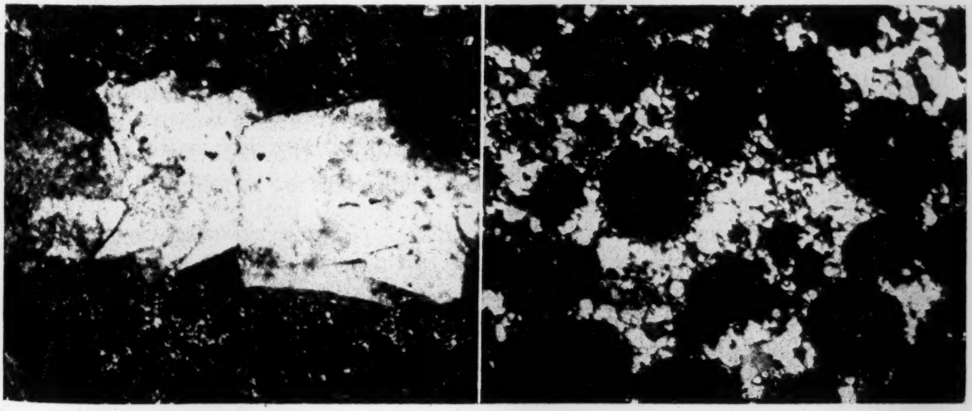
(a) +2.40 chert of class 3. Shows a seam of quartz (white) being replaced by brown iron oxide (black). The white of the groundmass is cryptocrystalline silica and the black is iron oxide and carbonaceous matter. Parallel nicols.

(b) +2.40 chert of class 3. Shows a seam and area of amorphous iron oxide (black) in a groundmass of cryptocrystalline silica (speckled gray). White areas are voids. Parallel nicols.



(c) +2.40 chert of class 3 Shows connected and isolated amorphous-iron-oxide filled voids (dark gray and black) in a groundmass of cryptocrystalline silica (light gray). Polished section in reflected light.

(d) −2.40 chert of class 5. Shows open voids (white) in a groundmass of indistinct fossils replaced by cryptocrystalline silica (mottled gray), amorphous iron oxide and carbonaceous matter (black). Parallel nicols.



(e) +2.40 chert of class 4. Shows large crystals of calcium carbonate (white) in a cryptocrystalline silica groundmass. Crossed nicols.

(f) +2.40 chert of class 6. Shows fine textured cryptocrystalline oolites (rounded speckled black areas) in a dense coarser cryptocrystalline groundmass. Crossed nicols.

FIG. 4.—Chert in Gravel (× 100).

are presumably present in the amorphous iron constituent since the cryptocrystalline silica groundmass is in every way similar to that of pebbles which develop no coating of air bubbles when preparing them for thin sections.

The greatest part of the iron constituent in all of the cherts examined was present as an amorphous hydrate which contains a variable amount of adsorbed and capillary water. The mineral is variable in composition and may range from the crystalline monohydrate goethite ($Fe_2O_3 \cdot H_2O$) through a number of amorphous forms, including limonite, to the rather vague species limnite ($Fe_2O_3 \cdot 3H_2O$). Therefore, a seam or group of connected voids "sealed" by this constituent might become potential reservoirs for adsorbed and capillary water. When such seams reach to the outside of the pebble, they might be expected to act as wicks and a means by which water can be taken up, or given up, depending on the temperature variations and moisture conditions surrounding the pebble. Freezing and thawing of such moisture within the seams would eventually cause rupture of the cryptocrystalline structure and allow a progressive ingress of capillary water to the more internal amorphous-iron-oxide-filled voids until an entire rupture of the pebble occurred under freezing conditions. The action would be similar in every way to that in which water filled open voids or pores and the final result would probably be the same although somewhat slower in developing.

Chemical analysis of this type of pebbles showed them to have as high as 6.4 per cent iron oxide, reported as Fe_2O_3, as compared with a satisfactory +2.50 chert from another district having 1.9 per cent iron oxide. The absorption of these pebbles (2.8 per cent) was also the highest of any of the 2.50 cherts studied.

The chemical reaction noted by Stanton[6] between opaline cherts and high-magnesian limestone with high-alkali portland cements was not observed in any of the cherts investigated. The test used for this potential reaction was the immersion of whole pebbles, and of fractured and pulverized specimens of the chert in 10 per cent solutions of sodium hydroxide for several weeks. The opaline silica in the rocks referred to by Stanton was readily dissolved and disintegrated by this test.

The conclusion from the microscopic observations was that in a majority of cases the variation in bulk specific gravity of the chert pebbles was due to variations in the relative abundance of the minute sealed and unsealed pores which are present in the cryptocrystalline groundmass. In the minor cases, where the amorphous iron oxide content is a factor in filling or sealing voids and fractures, the high specific gravity of this mineral may outweigh the buoyancy of the submicroscopic pores so that the pebble becomes classified in the 2.50 group. Absorption tests may be necessary to differentiate these pebbles from the satisfactory dense 2.50 cherts.

A microscopic classification of the cherts encountered in this investigation is given below. Only one of the classifications was not basically cryptocrystalline silica. All of the classifications are subject to variations and an estimate of durable quality should be based upon the flotation test.

The classes are as follows:

Class 1. Coarse-Textured Cryptocrystalline Chert:

These rocks were generally a light brown to buff or light gray color. They were very hard, dense, usually of vitreous luster, and broke with a distinct conchoidal fracture. Voids and pores were usually absent or negligible in the cherts having a bulk specific gravity of +2.40. In polarized light, under the petrographic microscope, they showed a relatively uniform, dense, coarse-textured cryptocrystalline structure composed predominantly of quartz with minor amounts of chalcedony, amorphous and crystalline iron oxide, calcium carbonate, and carbonaceous matter, (Fig. 3(*a*) and (*b*)). Pebbles of this type having a bulk specific gravity lower than 2.40 have a porous structure, as illustrated in Fig. 3(*f*), which would make them unsatisfactory as aggregate.

Class 2. Fine Textured Cryptocrystalline Chert:

This type of rock was similar in general appearance to class 1, but appeared to have a little more vitreous luster on freshly broken surfaces. In polarized light, under the microscope, it had a much finer textured cryptocrystalline structure than class 1 with a composition almost entirely of quartz with a minor amount of impurities. This type of chert may vary from a distinctly porous mass to one almost devoid of pores. In general this type of chert would usually make good aggregate when properly classified by the flotation test (Fig. 3(*c*), (*d*) and (*e*)).

Class 3. Iron Oxide and Hydroxide-Bearing Chert:

While iron oxide and iron hydroxide are usually present in most cherts this classification is used to designate those cherts in which the iron oxides are a major constituent of the rock. This type of rock is usually a very dark brown color throughout, with a dull luster on broken surfaces. Streaks and patches of various shades of brown are common. Although these pebbles often appear to be dense and hard they frequently break along fractures or seams. Under the microscope the cryptocrystalline silica structure is usually similar to classes 1 or 2, but, in addition, there is an unusual amount of amorphous iron oxide. A chemical analysis of a sample of this chert showed an iron oxide content of 6.4 per cent as compared with 1.9 per cent for a more durable and satisfactory brown colored chert. Because of the possibility of the presence of seams, cracks or voids sealed with a form of iron oxide, which may have an affinity for adsorbed water, pebbles of this classification should be questioned as durable aggregate although not all pebbles of this type were unsatisfactory (Fig. 4 (*a*), (*b*), (*c*) and (*d*)).

Class 4. Calcium Carbonate Bearing Chert:

Although calcium carbonate is present in most cherts in minor quantities, this classification is used to designate those cherts in which the calcium carbonate content is sufficient to be considered as one of the major constituents of the rock. This type of rock is usually a gray or light buff color and is often somewhat softer than classes 1 and 2. Under the microscope, the texture of the cryptocrystalline silica may be similar to either class 1 or 2, but, in addition, it contains an appreciable amount of calcite ($CaCO_3$) intimately associated with the cryptocrystalline groundmass. Such pebbles often contain sufficient carbonate to effervesce readily in cold dilute hydrochloric acid. This class of chert when free of voids should make satisfactory aggregate in any construction for which good limestone could be used (Fig. 4(*e*)).

Class 5. Fossiliferous Chert:

Chert pebbles containing an abundance of replaced fossils whether visible to the naked eye or not, and whether replaced by quartz, chalcedony, or forms of iron oxide are included under this classification. The fossils are embedded in a cryptocrystalline groundmass of silica. In the dense, hard varieties, iron oxide and carbonaceous matter often constitute a notable proportion of the mineralogical content of the pebbles giving them a dark color. Very porous specimens, on the other hand, may be very nearly white. In general this type of chert should be viewed with suspicion as aggregate and should only be used as such after it has been found to pass the flotation test requirements. While many of the fossils in this type of chert have been completely replaced by quartz or chalcedony forming a dense compact mass, many have not been completely replaced and there may be an abundance of open or amorphous-iron-filled voids which would act as potential reservoirs for water and its destructive effects on freezing (Fig. 4 (*d*)).

Class 6. Oolitic Chert:

To the casual observer, this type of chert may often appear similar to ordinary brown chert, but on breaking a pebble it is found to consist of innumerable minute oolites. Under the microscope, the oolites in some cases proved to be very fine textured cryptocrystalline silica bonded by a coarser textured cryptocrystalline structure of silica (Fig. 4 (*f*)). In other cases, they proved to be minute rounded quartz sand grains bonded by cryptocrystalline silica (Fig. 5). Strictly speaking, this latter form would not be a chert. However, the pebbles of this type encountered in this investigation had all the outward appearances of oolitic chert and would easily be selected as such by the macroscopic observer.

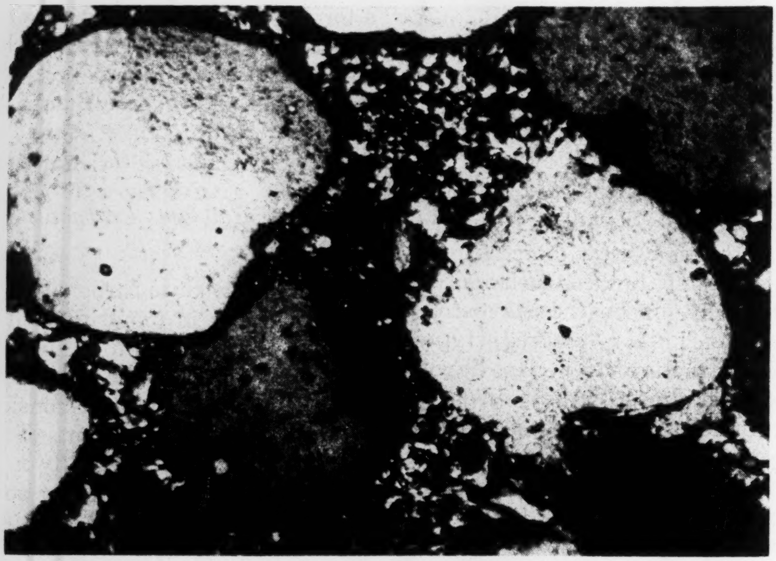


FIG. 5.—Quartz Sand Grains Cemented by Cryptocrystalline Silica. Crossed Nicols (× 100).

Class 7. Amorphous (Opaline) Chert:

This type of chert was not found in any of the gravels listed in Table I, but a sample was obtained from California for purposes of comparison. This sample was a dull gray color streaked with brown. It was relatively soft and could be scratched with a knife blade, although it broke with a semi-conchoidal fracture. Under the microscope it was found to consist almost entirely of amorphous silica in which numerous distinct fossils were embedded (Fig. 6). In polarized light this type of chert did not show the characteristic cryptocrystalline structure possessed by the cherts studied for this investigation, but remained dark except for the scattered fossils replaced by crystalline silica. This material has the relatively low specific gravity of 2.10 and would emphatically fail to pass the flotation test. Because of the nature of this type of chert and the presence of innumerable visible fracture cracks, which allowed pieces to be broken off by hand, this material would be expected to make very poor aggregate in concrete.

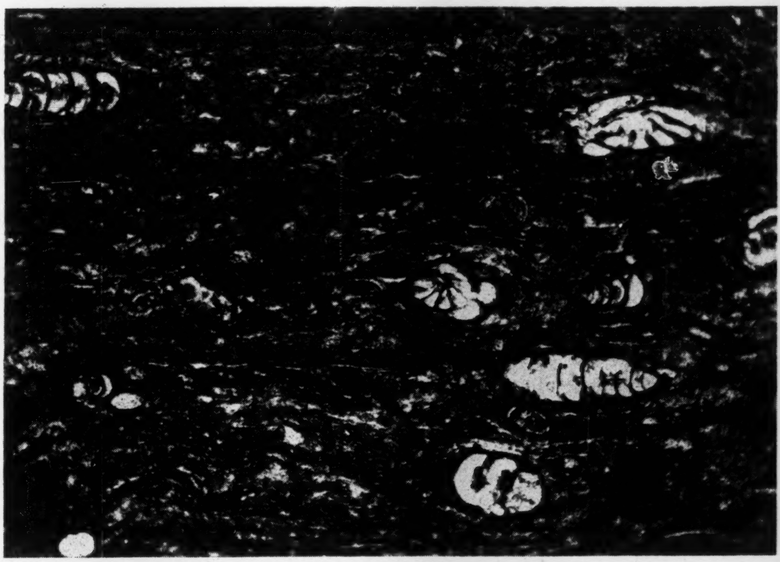


FIG. 6.—Amorphous (Opaline) Silica Containing Fossils Replaced by Crystalline Silica. Parallel Nicols (× 100).

DISCUSSION

The tests and observations conducted during the investigation indicate that attempts to differentiate by visual examination between sound and unsound varieties of cherts found in gravels, obtained from the regions included, is unsatisfactory and generally impracticable of achievement, because the factors which influence durability are too frequently undiscernible to the eye or by low-power lenses. The color of the chert pebbles may have some relationship to durability but it is too vague for practical use.

Quantitative analysis of a representative sample of aggregate by an absorption test alone is not a practical or desirable procedure, because: (1) the presence of a relatively small, but objectionable, quantity of unsound chert would not be detectable in a gravel sample of basically low absorption, (2) materials other than chert may be present which have a relatively high absorption and yet may be sound in concrete, and (3) the absorption method does not lend itself well to adjustment in tolerable limits in specifications for structures of varying surface area – mass ratio or climatic exposure.

Determination of the durable quality of chert contained in aggregate by means of a freezing-and-thawing test of the aggregate in concrete is unsatisfactory, particularly because there is no standard for such a test and the establishment of such a standard in the near future is unlikely. Furthermore, results of a freezing-and-thawing test are difficult to express quantitatively.

A freezing-and-thawing test of the unconfined aggregate, as a means of determining the soundness of the chert content, is not satisfactory for the reasons stated above and because it would be influenced by the relative durability of the other minerals present.

TABLE VII.—INFLUENCE OF TIME OF BOILING AND EVACUATION ON THE ABSORPTION AND BULK SPECIFIC GRAVITY OF SAMPLE W-1.

Bulk Specific Gravity	Material	Basis of Separation									
		5-hr. Boiling		5-hr. Evacuation		10-hr. Boiling		15-hr. Boiling		36-hr. Evacuation	
		Percentage of total	Absorption, per cent	Percentage of total	Absorption, per cent	Percentage of total	Absorption, per cent	Percentage of total	Absorption, per cent	Percentage of total	Absorption, per cent
+2.50.......	Chert	41.0	0.9	36.1	0.9	38.0	1.1	38.5	1.0	39.5	1.0
	Nonchert	44.8	1.7	40.2	1.7	40.0	0.8	42.0	1.2	42.7	1.3
−2.50.......	Chert	4.8	3.5	12.6	3.4	8.5	3.1	7.5	3.3	7.6	3.5
+2.40.......	Nonchert	3.2	3.9	3.2	3.7	4.8	3.7	3.6	3.8	3.6	3.9
−2.40.......	Chert	4.0	5.6	2.9	5.8	4.2	4.9	4.1	5.7	3.2	5.4
+2.30.......	Nonchert	0.5	6.8	3.2	6.8	2.6	6.3	1.9	5.9	1.6	9.1
−2.30.......	Chert	1.5	9.0	1.8	8.7	1.3	9.0	2.3	7.1	1.6	9.6
	Nonchert[a]	0.2	12.0	0.0	...	0.6	12.0	0.1	16.9	0.2	9.5

[a] The nonchert lighter than 2.40 in this sample was an argillaceous shale.

The magnesium sulfate soundness test provides a better and more reproducible means for testing the durability of unsound chert than does the freezing test. But, it is incapable of separating the unsound and the sound cherts. Many unsound cherts disintegrate differently and more slowly in the sulfate test than do most other unsound aggregates.

Microscopic analysis, competently performed, is an excellent method for differentiating between sound and unsound chert, but only on an academic basis. The use of a polarizing microscope would be impractical as a means for testing aggregate in the field. However, the microscopic studies proved that the unsoundness of the cherts investigated was due principally to the nature and extent of the void structure of the rock.

The 5-hr. boiling period was chosen for use in the absorption, bulk specific gravity and flotation tests for two reasons: (1) this period permits the accomplishment of the entire flotation test during an 8-hr. day in the field, and (2) results of longer boiling periods and periods of evacuation developed no significant changes or improvements especially in the low gravity particles. A typical example of these influences on parallel samples of gravel W-1 is given in Table VII. The variations in the data are due undoubtedly to lack of perfect duplication of mineral distribution in each of the parallel samples.

The flotation procedure developed by this laboratory misses practical perfection, as a mechanical means of determining quantitatively the amount of unsound chert in aggregate, to the extent that there are some varieties of unsound fractured chert containing relatively high percentages of iron which would not be detected by the test. There is also the fault that material other than unsound chert will float in a 2.40 solution of bromoform. Lignite, wood, coal and highly absorptive argillaceous shale are the materials of this type, found in the samples tested, which are readily identifiable and present no practical obstacle to the use of the test. However, a specie of sandstone was observed in one gravel sample which was very difficult to identify macroscopically as nonchert. This sandstone and the argillaceous shale were extremely absorptive. The sandstone had an absorption of 10.5 per cent and the shale had an absorption of

TABLE VIII.—DISTRIBUTION OF CHERT AND NONCHERT IN GRAVEL SAMPLE O1A.

Bulk Specific Gravity	Distribution by Flotation, per cent	Chert, per cent	Nonchert, per cent	Classification of Noncherts	
				Carboniferous, per cent	Porous Sandstone, per cent
+2.50	79.2		Not separated		
2.40 to 2.50	12.4	10.2	2.2	0	2.2
2.30 to 2.40	6.1	5.1	1.0	0	1.0
−2.30	2.3	2.0	0.3	0.1	0.2

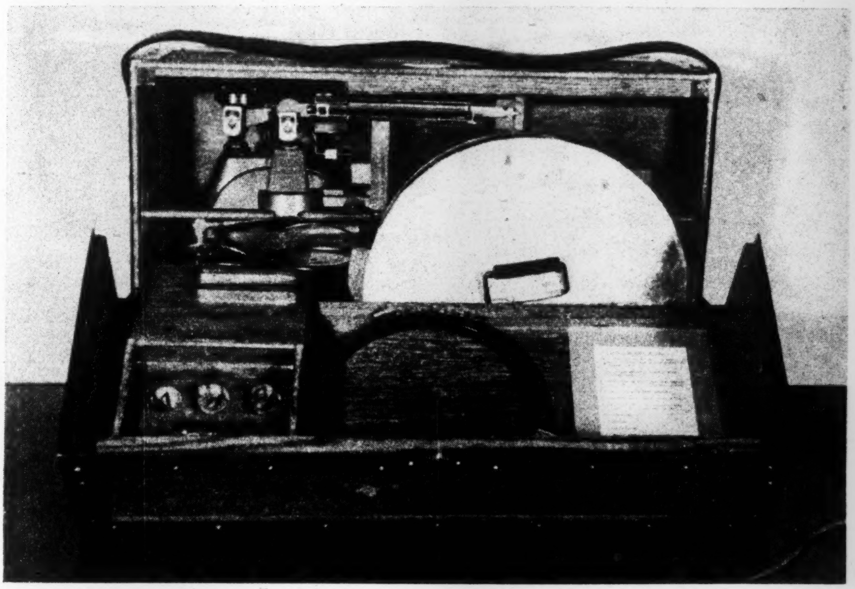


FIG. 7.—Flotation Field Kit. Showing entire equipment packed in kit for transportation.

Carrying case.—Constructed of oak plywood with copper strip protection along corners and edges. With lock and leather carrying handle. Interior constructed with shelves, inserts and containers for the secure holding of parts during transportation. Weight—31.00 lb.

Flotation solution.—500 ml. of bromoform—mono-bromo-benzene with specific gravity desired—usually 2.40. In glass-corked bottle. Weight full—5.30 lb.

Diluent.—500 ml. of mono-bromo-benzene. Specific gravity 1.46. In glass-corked bottle. Weight full—4.20 lb.

Hydrometer.—In carton. Range 2.00 to 3.00. Calibrated in increments of 0.01 and accurate to 0.02 specific gravity. Weight 0.40 lb.

Dipping spoon. Wire mesh spatulate end on brass rod 8 in. long. Weight 0.10 lb.

Flotation container.—500 ml. conical glass graduate. Weight—1.50 lb.

Boiler.—Circular metal pan with cover of sufficient capacity to contain 20 lb. of pebbles plus sufficient water to cover sample during boiling period. Weight -1.80 lb.

Tripod. -Circular metal tripod to support boiler over gasoline burner. Weight –2.20 lb.

Burner.—Gasoline -Barthol type. Weight—1.50 lb. A substitute may be used, but the small size, efficiency and light weight of this burner is advantageous.

Fuel Can.—1-gal. tin can for gasoline fuel supply. Weight—0.30 lb.

Scale.—Eimer & Amend No. 17026 Solution Scale Triple Beam Balance (Ohaus) or equivalent with two 1000-g. and one 500-g. extra weights.

Cans. -Two ½-gal. paint cans for separated samples after flotation. Weight 1.20 lb.

Rags.—Rags or towels for hand drying boileds amples and for packing around equipment to eliminate chances of breakage in kit during transportation. Weight—1.00 lb.

12.3 per cent. The structure of these materials contained a network of large open pores and numerous unsealed voids. The large amount of water taken up by these pores during the 5-hr. boiling period provided a false bouyancy to the pebbles when introduced into the heavy gravity bromoform solution which is immiscible with water. Both materials would make most unsatisfactory aggregate for concrete. A typical example of the flotation of non-cherts is given in Table VIII.

In spite of the defects described above, the flotation test affords a simple, rapid, mechanical field means for the separation of a majority of the unsound chert in concrete aggregate. The equipment required is simple, rugged and can be packed in a compact kit-box (Figs. 7 and 8) for transportation and use anywhere in the field.

Reference to the durable quality of cherts in terms of bulk specific gravity would do much to alleviate the confusing suspicion which now surrounds the material as a whole. Also, reference to relative durability in such terms would permit the use of quantitative limits in specifications which would be suitable for individual projects. The limits could be variable in terms of gravity and per cent.

From a field survey made recently, it may be estimated that use of the flotation test and a bulk specific gravity of 2.40 would insure the detection of not less than 75 per cent and, in most cases, up to 90 per cent of the unsound chert in the aggregate. The use of a higher separation gravity would probably introduce too many variables by including large percentages of nonchert rocks.

Various investigators have reported that the degree of natural saturation of unsound chert pebbles has an important influence on the durability of the chert in concrete. Although this condition was not investigated specifically in this program, the theory is reasonable and compatible with the data presented herein.

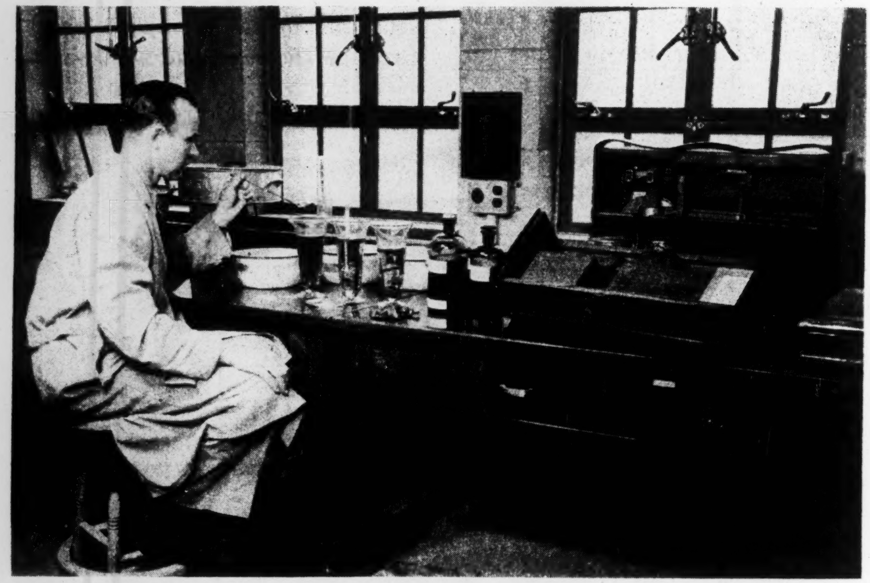


FIG. 8.—Flotation Field Kit. Showing equipment in use.

Conclusions

The conclusions which appear to be indicated by the tests described herein are as follows:

1. The cherts in the regions investigated were composed basically of cryptocrystalline silica with varying contaminations by the oxides and hydroxides of iron, calcium carbonate, clay and carboniferous matter. Relatively little chalcedony and essentially no amorphous (opaline) silica were observed.

2. Classification of cherts by use of the polarizing microscope is excellent for use in a research laboratory, but not practical as a means for field or routine testing of aggregate.

3. The accelerated physical tests such as freezing and thawing, magnesium sulfate and absorption are useful, but not satisfactory for judging the quantitative presence of unsound chert.

4. The bulk specific gravity appears to be closely related to the absorptive capacity of chert which appears to be the principal index of chert durability.

5. The separation of the unsound from the sound varieties of chert by means of the flotation test appears to be a reasonably reliable, rapid, simple, mechanical means for use in field determinations.

Acknowledgment:

The investigations reported were conducted as a research project of the Central Concrete Laboratory of the North Atlantic Division, U. S. Engineer Dept.

The authors are indebted to M. R. Smith, Sam Goldman and Joseph Bayer of the laboratory for their assistance in conducting the physical tests.

Acknowledgment is made to the U. S. Engineer offices at Vicksburg, Miss., and at Louisville, Ky. for providing a number of the gravel samples; to W. H. McAlpine and B. W. Steele of the Office of the Chief of Engineers and to Stanton Walker of the National Sand and Gravel Assn. for their helpful suggestions.

DISCUSSION

THE CHAIRMAN (*Mr. Stanton Walker*[1]).—I wish to comment, at this time, on only one of the points mentioned by Messrs. Wuerpel and Rexford. They stated that they had subjected several cherts, both in the original solid form and in powdered form, to 10 per cent solutions of sodium hydroxide at room temperature and did not find them to be attacked. At the National Sand and Gravel Association Research Foundation, at the University of Maryland, we have been devoting a great deal of attention to chert gravels and we have been able to break them down by immersing them in sodium hydroxide solutions of various strengths up to 10 per cent. The rate of "digestion" is greatly affected by the temperature of the solution, as well as by the strength, it being extremely slow at room temperature and quite rapid at elevated temperatures. Just what the significance of the results is I do not know, but there appears to be some evidence to indicate that there is a vague relationship between the rapidity with which the different cherts are digested and their service records, the cherts which are slowly attacked by the sodium hydroxide having the better service record.

Details of certain of our results are contained in the following discussion by Mr. Bartel.

MR. FRED F. BARTEL[2] (*by letter*).—Tests carried out at the National Sand and Gravel Association Research Foundation at the University of Maryland afford data which supplement the findings of Messrs. Wuerpel and Rexford. The authors point out that they investigated the chemical reaction between sodium hydroxide and the cherts which they studied, using a 10 per cent solution at room temperature and following the procedure described by Stanton[3] of the California Highway Dept. They found no reaction for either the chert pebbles or for material which had been pulverized.

TABLE I.—DISINTEGRATION OF VARIOUS AGGREGATES BY A 10 PER CENT NaOH SOLUTION.

Lot	Rock Type	Loss in Weight After 25 hr. at 212 F., per cent	Loss in Weight After 134 days at Room Temperature, per cent
No. 1249	Clear quartz	1.0	0
No. 1238	Clear quartz	1.4	0
No. 1249	Stained quartz	1.5	0
No. 1238	Stained quartz	0.9	0
No. 1021	Coarse grained sandstone	100	0
No. 1019	Coarse grained sandstone	30.5	5.3
No. 947	Limestone	0	0
No. 1081	Chert	100	4.1
No. 1062	Chert	100	4.6
No. 1220	Chert	100	9.7
No. 1027	Chert	57.5	5.6
No. 1207	Chert	100	1.0
No. 1119	Chert	100	3.8
No. 1234	Chert	100	2.0
No. 1034	Chert	100	1.6

Our work at the University of Maryland has involved studies of a number of chert gravels from long-established commercial sources. In exploratory tests, we have studied their reaction to sodium hydroxide solutions of various strengths, up to 10 per cent, and at temperatures ranging from room temperature to 212 F. Without exception disintegration has occurred for all of the cherts studied, although the rate of disintegration varied greatly, depending upon the quality of the material.

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[1] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

[2] Research Fellow, Stanton Walker Fellowship of the National Sand and Gravel Association Research Foundation, University of Maryland, College Park, Md.

[3] T. E. Stanton, "Expansion of Concrete as Influenced by a Reaction Between the Cement and Aggregate," *Engineering News-Record*, Vol. 124, No. 5, February 1, 1940, p. 59.

The accompanying Table I gives the results of some of the first tests and shows the effect on different rock types of exposure to 10 per cent solutions of sodium hydroxide at room temperatures and at 212 F. Each test result represents a single particle about 1 in. in size. In the case of the solution at room temperatures, no action was noted for several weeks and no measurements of weight were made during the early periods. After about four months, however, action was apparent and the weight losses shown in Table I were determined at 134 days. For the chert gravels they range from 1 to about 10 per cent; for the other rock types studied, with one exception, no disintegration took place. The exception was a coarse-grained sandstone with ferrous binder. Exposure for 25 hr. at a solution temperature of 212 F. resulted in complete breakdown of all of the chert gravels except one, and that lost more than one-half of its weight. Quartz and limestone aggregates were negligibly affected. Considerable breakdown occurred for the coarse-grained sandstone tested.

TABLE II.—EFFECT OF CONCENTRATION OF NaOH SOLUTION AT 212 F. ON RATE OF DISINTEGRATION.

Sample Lot 1081

Solution	Weight Loss After 25 hr., per cent
Water	0
2 per cent NaOH	8.6
5 per cent NaOH	100
10 per cent NaOH	100

In further exploratory tests, the effect of strength of solution was investigated at a temperature of 212 F. The exposure was for 25 hr.; it will be observed from the accompanying Table II that considerable disintegration was obtained with a 2 per cent solution.

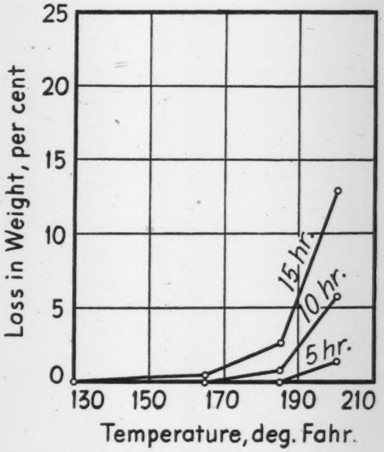

FIG. 1.—Effect of Temperature on Rate of Disintegration, Chert Gravel No. 1034 in 10 per cent NaOH Solution.

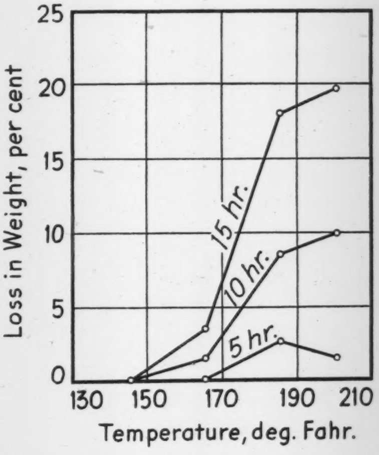

FIG. 2.—Effect of Temperature on Rate of Disintegration, Chert Gravel No. 1081 in 10 per cent NaOH Solution.

It is evident from Table I that the rate of disintegration is greatly affected by temperature of the solution. Accordingly, further tests were made at temperatures varying from 130 to 212 F.

The effect of exposure to a 10 per cent sodium hydroxide solution for 5, 10, and 15 hr. on two chert gravels is shown in the accompanying Figs. 1 and 2. No disintegration was found at 15 hr. until temperatures in excess of about 130 to 150 F. were maintained.

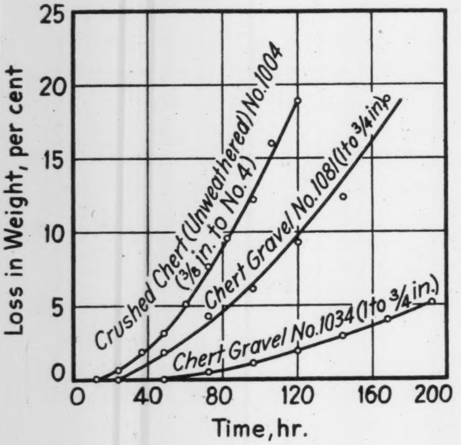

FIG. 3.—Effect of Time of Exposure on Disintegration, Cherts in 10 per cent NaOH Solution at 130 F.

Since 130 F. impressed us as being a significant temperature, one readily attainable in the surface of a concrete road slab, further tests were carried out for periods up to 192 hr. The data are shown in the accompanying Fig. 3. Two chert gravels from commercial sources, graded 1 to $\frac{3}{4}$ in. and one crushed unweathered chert (sometimes described as novaculite) graded $\frac{3}{8}$ in. to No. 4 were used. Reference to Fig. 3 will show that considerable disintegration occurred for all of the materials tested but that the rate varied greatly depending upon quality. It is probable that the results for the crushed chert (1004) are not strictly comparable with the others because of the smaller size used.

The accompanying Fig. 4 is of considerable interest since it suggests that time-rate-of-disintegration relationship curves for different temperatures are of the same shape. In other words, the periods required to produce a given disintegration vary linearly with the temperature. For example, in Fig. 4 it will be seen

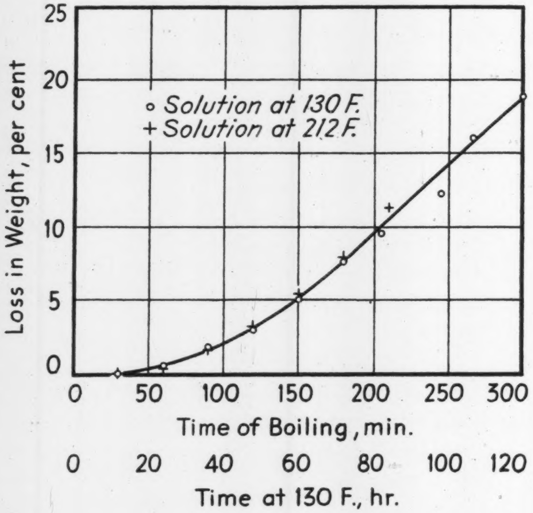

FIG. 4.—Relationship Between Temperature at Exposure and Rate of Disintegration, Crushed Chert (Unweathered) in 10 per cent NaOH Solution.

that for the single material tested it took 24 times as long to obtain a given disintegration at 130 F. as it did at 212 F. Other quite limited data indicate that this ratio of periods required at 130 F. to that required at 212 F. may be as low as 10 for certain of the materials which are attacked. Similarly, a comparison of data from Table I and Fig. 3 indicates that the time required to produce a given disintegration at room temperatures is 25 to 50 times that at 130 F.

Tests were also made to determine the distribution of particles by specific gravity on four chert gravels from commercial sources using a method similar to that of the authors. The data are shown in the accompanying Table III. Further studies of the characteristics of the materials in the different specific gravity brackets are under way.

TABLE III.—DISTRIBUTION OF PARTICLES OF CHERT GRAVEL BY SPECIFIC GRAVITY.
Samples sieved to pass 1-in. sieve and to be retained on ¾-in. sieve.

Lot	Per cent of Sample in Each Specific Gravity Range						
	2.00 to 2.09	2.10 to 2.19	2.20 to 2.29	2.30 to 2.39	2.40 to 2.49	2.50 to 2.59	2.60 to 2.69
No. 1026, per cent by weight......	0.2	0.7	4.7	20.5	26.9	39.3	7.7
per cent by number.....	0.2	0.8	5.2	22.2	26.2	38.2	7.2
No. 1034, per cent by weight......	0.0	0.1	1.4	7.6	34.0	53.6	3.3
per cent by number.....	0.0	0.3	1.7	8.5	33.0	53.3	3.2
No. 1062, per cent by weight......	0.3	6.6	20.5	28.4	28.5	15.5	0.2
per cent by number.....	0.4	7.3	21.7	29.3	26.5	14.6	0.2
No. 1081, per cent by weight......	1.9	5.9	24.2	24.0	28.4	12.0	3.6
per cent by number.....	2.3	7.2	24.5	24.0	27.6	11.0	3.4

If the reaction between aggregates and sodium hydroxide solutions is related to the action of aggregates in concrete made with high alkali cements, as Stanton[3] suggests, the results of these exploratory tests may be of considerable significance. However, it should be pointed out that our investigations have not gone far enough to permit us to say that such a relationship exists for the materials we are studying.

The work described herein was carried out under the general supervision of Stanton Walker. Much of it was started by M. Cedric Gleason of the laboratory staff and Mr. Gleason has cooperated in the continuation of the work carried out as part of the fellowship research.

MESSRS. F. V. REAGEL[4] AND T. F. WILLIS[4] (*by letter*).—The authors have performed a real service by bringing to the attention of engineers some of the recent work on the use of chert as coarse aggregate, also by their petrographic description of some of the cherts of the Mississippi Valley area, and by outlining a method for separating aggregate particles of different porosities. To many of us the chert problem is an old but still annoying question and anything which may assist in its answer is most welcome.

Recently the Research Division of the Bureau of Materials of the Missouri State Highway Dept. has inaugurated, as one phase of a general investigation, tests along the lines described by Messrs. Wuerpel and Rexford. A few data are available which may be of interest as a complement to their work. The initial results presented here are based on tests of a chert gravel coarse aggregate which has a poor service record in concrete pavements. The aggregate as produced for concrete pavement has an average bulk specific gravity of *2.51* and, in the stream-wet condition, 3.8 per cent absorbed moisture. By means of high gravity liquids (various mixture of carbon tetrachloride, specific gravity *1.59*, and acetylene tetrabromide, specific gravity *2.95*) and a technique essentially similar to that described in the paper, the saturated aggregate was separated into three fractions of different bulk specific gravities, namely, less than 2.4, 2.4 to 2.5, and over 2.5.

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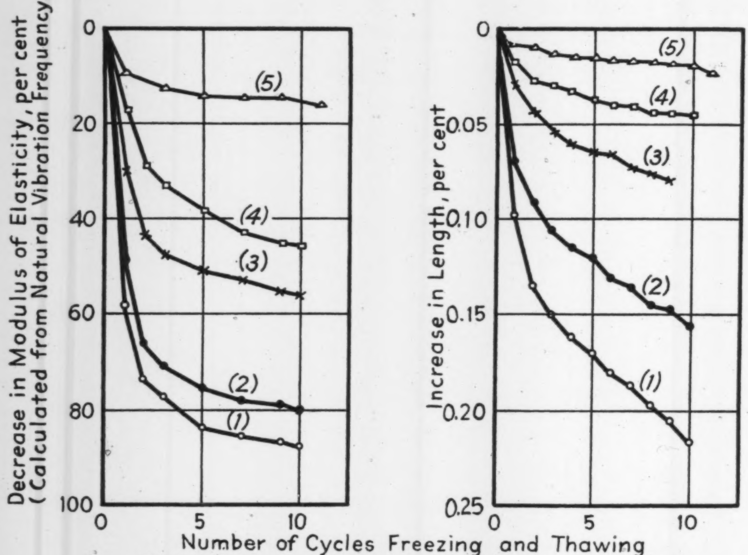
[4] Engineer of Materials, and Chief of Research Division, respectively, Bureau of Materials, Missouri State Highway Dept., Jefferson City, Mo.



Each point represents average of results on 3 specimens

Legend	Aggregate	Bulk Spefic Gravity: Range	Bulk Spefic Gravity: Average	Concrete: Slump, in.	Mix	W/C	Size of Specimen, in.	Curing
—×—×—	No. 1	-2.4 +2.5	2.51	$3\frac{1}{4}$	1:2.88: 4.15 by abs. vol.	0.77	Width $3\frac{1}{2}$ Depth $4\frac{1}{2}$ Length 16	7 days moist room, 21 days in water at 70 F.
—○—○—	No. 1	-2.4	2.34	$4\frac{1}{2}$				
—●—●—	No. 1	+2.4 -2.5	2.48	4				
—□—□—	No. 1	+2.5	2.58	$5\frac{3}{4}$				
—△—△—	No. 2		2.65	2				

FIG. 5.—Resistance to Freezing and Thawing of Concretes Made with Chert Gravel Aggregate of Different Bulk Specific Gravities.

These three gravity fractions, similarly graded from $1\frac{1}{4}$ to $\frac{1}{4}$ in., were incorporated in three batches of concrete all of which contained the same mortar. From the concrete $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16-in. beams were fabricated. When 28 days old, the beams were subjected to consecutive cycles of freezing in air and thawing in water. The temperature-time record of the cycle used is shown in the accompanying Fig. 6.[5] Tests on the concrete specimens were made immediately prior to the initial cycle of freezing and thawing and at the end of 1, 3, 5, 7, 9, and 10 cycles. These tests consisted of the determination of the modulus of elasticity by the dynamic method[6] and of the specimen length by means of 10-in. Whittemore strain gage.

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[5] As a sideline to this discussion, the writers cannot pass up the opportunity of stressing their idea of the importance, in reporting any results of tests involving freezing and thawing, of a full description of the freezing-and-thawing cycle used.

The results are shown in the accompanying Fig. 5, in which the percentage decrease in elastic modulus and the percentage increase in length are plotted against the number of cycles of freezing and thawing. For comparison, the results on concrete made from the stream-wet aggregate containing all the gravity fractions in the proportions in which they naturally occur (curve 3), and also from aggregate No. 2 (curve 5) a non-chert which has an excellent service record, are also plotted.

The graphs show that the deterioration of the various concretes under the accelerated freezing-and-thawing cycles used was qualitatively the same regardless of whether the deterioration[7] was measured by "decrease in elastic modulus" or "increase in length." These and numerous other tests have convinced us that, when concrete deterioration under freezing and thawing is due to an unsound aggregate, either test is an excellent criterion of the relative deterioration of different concretes.

It is evident from the graphs that, for the particular aggregate employed, the resistance to the accelerated freezing-and-thawing cycle used is much greater for concrete containing the aggregate fraction of greatest bulk specific gravity than for that containing aggregate fractions of lower gravity. It is also indicated that removal of the low-gravity aggregate fractions (less than 2.5), of which there are approximately *43.0* per cent in the stream-run material, and use of only the highest gravity fractions (over 2.5) produced concrete that was more resistant than that in which the stream-run gravel was used. However, the difference in the resistance to freezing and thawing in these two cases (curve 4 *versus* curve 3) is not nearly so great as it is between concretes made from the two lower gravity fractions and that made from the highest gravity fraction (curves 1 and 2 *versus* curve 4). In other words, removal from the stream-run material of all the aggregate having a gravity less than 2.5 caused only slight improvement in the resistance of concrete to freezing and thawing, but use of only the lower gravity fractions as aggregate resulted

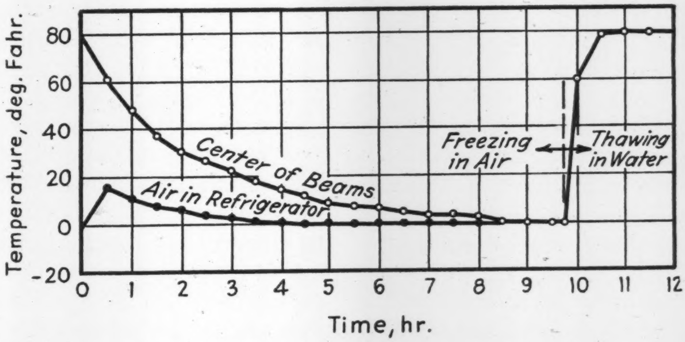

FIG. 6.—Typical Freezing-and-Thawing Cycle.

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[6] For a complete description of the apparatus used see discussion by Willis and De Reus of the paper by W. T. Thomson, p. 1123.

[7] For discussion and illustration of the relation between concrete deterioration as measured by "decrease in elastic modulus" and "decrease in flexural strength" see p. 1127 and Fig. 3 of the discussion by Willis and De Reus of the paper by W. T. Thomson.

in a material decrease in the resistance of the concrete.

One of the principal points brought out by the tests is that concrete made from even the highest gravity fraction (all particles over 2.5 and an average bulk specific gravity of 2.58) of the aggregate having a poor service record showed considerably less resistance to freezing and thawing than that of the non-chert aggregate which has a good service record (compare curves 4 and 5). Wuerpel and Rexford state that the high gravity fraction of one of the aggregates they tested also showed considerable deterioration. They explain this on the basis of the occurrence of particles that contained seams filled or sealed with amorphous iron oxide. The iron, having a higher density than the chert, caused the particles to fall in the high gravity fraction despite the fact that they were relatively porous and absorptive. While we have made no petrographic examination of our aggregate, there is no macroscopic evidence that such a condition exists; nor does the relative absorption of the different fractions indicate this to be the case. Rather, we fear, our results indicate that the degree of porosity of a particle is only one of the conditions which delineate the durability of the particle. If this be true, mere knowledge of the bulk specific gravity of aggregate particles will not give evidence of their relative durability. Other investigators have pointed out that the size of the pores and their degree of saturation are probably as important as the total porosity in influencing the resistance of aggregate to freezing and thawing. Furthermore, it is entirely possible that chert or any other aggregate particles, even though having a very low porosity may cause concrete, in which it is used, to have a poor resistance to freezing and thawing. We are thinking particularly of aggregate particles which, due to the conditions under which they were formed and to the orientation of the mineral grains, may be under considerable internal stress.

By the foregoing, we do not intend to condemn the gravity test as a criterion of the relative durability of aggregate particles but do suggest that its implications, with respect to an aggregate whose service record is not known, should be carefully considered.

The authors state that determination of the durability of chert contained in aggregate by means of a freezing-and-thawing test of the aggregate in concrete is unsatisfactory. They cite as reasons for this statement that there is no standard for such a test and that results of a freezing-and-thawing test are difficult to express quantitatively. Their first reason could be applied to any of the so-called soundness or durability tests and, therefore, should not serve as a specific condemnation of the freezing-and-thawing test. As for their second reason, it has been our experience that both the "loss in flexural strength" and the "loss in the elastic modulus" have very promising possibilities as methods of determining and quantitatively expressing the effect of freezing and thawing. Admittedly, there is need for further study of the variables involved in the freezing-and-thawing test and for closer control or elimination of certain of these variables. We would strongly advocate that the proper committees carry on such a study in the near future lest dissatisfaction, such as the authors express, become general and cause premature abandonment of the test.

The authors also express their dissatisfaction with the freezing-and-thawing test as applied to unconfined aggregate for the same reasons cited against the freezing-and-thawing test of the aggregate in concrete, and also because they say the results would be influenced by the relative durability of minerals other than chert which might be present. Based on our experience, it is our belief that the A.S.T.M. Tentative Method of Test for Soundness of Aggregates by Freezing and Thawing (C 137 – 38 T)[8] might, with a few modifications, be satisfactory. With regard to the influence of extraneous minerals on the results of this test, we fail to see that this is a point in question. Certainly the prime purpose of a durability test applied to any aggregate is to determine the durability of that aggregate as a whole, regardless of whether it be made up of one or a number of minerals.

In discussing the magnesium sulfate test, the authors state that this test provides a better and more reproducible means for testing the durability of unsound chert than does the freezing-and-thawing test but that it is incapable of separating the unsound and sound cherts. From our experience with the freezing-and-thawing test, we would suggest that if, in making this test, the samples were subjected to a vacuum treatment and permitted to absorb the magnesium sulfate solution while under vacuum, it is possible that more concordant results might be obtained. However, we cannot agree that the magnesium sulfate test has any merits which are not possessed by the freezing-and-thawing test. In fact, in our opinion it is subject to all the objections that pertain to the freezing-and-thawing test and, in addition, is completely artificial, reproducing in no known way any of the actual weathering to which aggregate is subjected.

There is one point regarding the so-called flotation-separation method which should be called to the attention of users, namely, that the greater the proportion of the total pore space filled with water prior to flotation, the higher the gravity group into which a particle will fall. In other words, if two particles have equal density and porosity but the first has a larger proportion of impermeable pores, it will fall in a lower gravity group than the second. If Messrs. Wuerpel's and Rexford's conclusion that "the absorptive capacity of chert which appears to be the principal index of chert durability" is correct, the flotation test may under certain circumstances give misleading results, that is, in the case of porous particles which do not absorb water.

MESSRS. CHARLES E. WUERPEL[9] and E. P. REXFORD[9] (*Authors' closure by letter*).—In reply to Mr. Stanton Walker's comments on the disintegration of chert by sodium hydroxide, we wish to point out that during our study of chert we had immersed a number of chert pebbles in a 10 per cent solution of sodium hydroxide for several weeks at room temperature. At the time of writing the paper no apparent attack on the chert pebbles could be observed. This fact is borne out by Mr. Bartel in his discussion of our paper wherein he found that "no action was noted for several weeks" when the cherts were subjected to room temperature.

This test had been made by the authors simply to obtain a comparison between the reactivity of the California chert, as reported by T. E. Stanton, which we also found to disintegrate readily in the sodium hydroxide solution, and those cherts which we were investigating. No complete study of the reactions between chert and sodium hydroxide was attempted because it was considered to be beyond the scope of the present paper.

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[8] 1939 Book of A.S.T.M. Standards, Part II, p. 990.

[9] Engineer-in-Charge, and Associate Petrographic Microscopist, respectively, Central Concrete Laboratory, North Atlantic Division, U. S. Engineer Dept., U. S. Military Academy, West Point, N. Y.

Most of the pebbles which we had immersed in the sodium hydroxide solution were thrown away as soon as it was definitely shown that no disintegration such as that which is experienced with the California opaline cherts was to be encountered with the cherts under investigation. However, a few pebbles were left in the solution up to the present time, about six months, and although a sediment in the bottom of the bottle indicates that some reaction has occurred the pebbles themselves are still as hard as when they were first placed in the solution. We mention this with the realization that it gives very little scientific information other than to show that some pebbles of chert may remain hard and apparently durable for extended lengths of time in a 10 per cent sodium hydroxide solution at room temperature.

Mr. Bartel is to be commended on his investigation of the reactions between different rock aggregates and sodium hydroxide solutions. Such a study is of fundamental importance. We question, however, the advisability of selecting a temperature above that of the higher climatic temperatures as the significant temperature to investigate, unless, of course, that temperature is to be considered only as giving a possible accelerated service condition. While 130 F. may readily be obtainable in a concrete road slab, it seems unlikely that such a temperature would persist under the moisture conditions necessary to cause a chemical reaction between the relatively small alkali content of the cement and a reactive aggregate.

While it is assumed that this study was made with the idea of investigating the detrimental effects on the aggregate particles of alkalies present in cement, it might be expected to be of still further significance when applied to alkalies present in the curing water and in possible ground waters with which the concrete may come in contact. Along this same line the study of the effects of carbon dioxide, taken from the atmosphere by rains, might prove to be of considerable value. Carbonic acid is known to play an important role in the natural disintegration of many rocks.

The discussion by Reagel and Willis is appreciated by the authors. While, in general their observations are concordant with those set down in the original paper, various divergencies of opinion exist, as would normally be expected. In reply to their statement that a concrete made of the highest gravity fraction of a poor service cherty aggregate had shown a lower resistance to freezing and thawing than had a concrete made with a nonchert aggregate with a good service record, we should like to point out that it was not our intention to imply that cherty gravel should be expected to make as good an aggregate as some of the best nonchert gravels. We did, however, attempt to show that many cherts can make satisfactory aggregate, and thus many otherwise satisfactory gravels having a moderate amount of chert need not necessarily be condemned because of its presence, as is often the tendency at the present time.

Furthermore, it is not quite clear in the discussion as to whether the highest and lowest gravity fractions mentioned therein apply to bulk specific gravity fractions of chert or of the aggregate as a whole. If the latter is the case, it is conceivable that the flotation procedure, as described in our paper, might encounter difficulties. The selection of 2.4 bulk specific gravity for the dividing line between good and bad chert was based on the action of chert pebbles, above and below that gravity, in freezing and thawing and in the magnesium sulfate test. Use of the flotation procedure to determine good from bad aggregate in other rock types would very possibly necessitate the use of a higher bulk specific gravity as the dividing line. Moreover, if the method of floating the total aggregate was used by Reagel and Willis in their experiment, there is a very good possibility that the nonchert pebbles were of inferior quality and were the cause of the relatively lower resistance to freezing and thawing and not the chert, as might be inferred from their discussion.

We agree that very often more complete tests, such as the freezing-and-thawing, the magnesium sulfate or a petrographic examination may be advisable and even necessary before a cherty gravel should be placed in service. As was pointed out in the original paper, cases can be encountered in which high gravity chert may fail in these tests. The paper offered an explanation, based on microscopic evidence, of one such failure. Such evidence could not be seen macroscopically as Reagel and Willis attempted to do.

Unquestionably the size of the pores and their degree of saturation is important in influencing the resistance to freezing and thawing. However, the fact remains that any pore or void capable of taking up and retaining water may be expected to develop internal stresses which can readily become sufficiently intense to cause rupture of the pebbles when under freezing conditions. In a sense the flotation test takes care of the question of pore size and degree of saturation in that it indicates whether or not sufficient total pore space is present to cause rupture when it is saturated under freezing conditions. The selection of a bulk specific gravity of 2.4 or above for satisfactory chert does not mean that a chert pebble of that gravity has no pores but that the pores present are sufficiently small and few so that the cohesive strength of the cryptocrystalline structure is sufficient to withstand the pressure exerted in freezing. Unfilled or partially filled pores would exert less pressure than completely filled pores because of the space available for the development of the ice crystals.

The effects of internal stresses in rock caused by the mode of formation or crystal orientation are, of course, well known, particularly as applied to igneous types of aggregate. It, however, has very little bearing on this paper.

In general the authors have no controversy with the freezing-and-thawing test nor do they suggest its replacement by the flotation method. It is frequently used in this laboratory, as also is a cathode ray oscillograph for measuring the "loss in elastic modulus." However, for the determination of the durability of chert pebbles, it has not been found practical or reliable at this laboratory. Subjecting the samples to a vacuum before freezing and thawing has not been attempted and it is very possible that more concordant results could be obtained by so doing. However, it is a time-consuming method requiring considerable apparatus before usable results can be obtained, and it is, therefore, a definite laboratory procedure. The flotation test, on the other hand, was introduced with the idea of offering a relatively quick, easy and inexpensive method of determining in the field whether a cherty gravel should be summarily condemned because of its unsound chert content or whether more extensive and expensive tests were warranted.

The point mentioned by Reagel and Willis concerning the possibility of error being encountered by the fact that two pebbles having equal density and porosity may differ in the degree to which they absorb water, does not seem to be of practical significance. The pebble which did not absorb water would of course be the lighter of the two in weight and would be classified in the lower gravity brackets by the flotation test, as it should be. The other pebble while heavier than the first could not possibly be expected to attain sufficient weight, by absorbing water with a specific gravity of 1.00, to sink in a liquid with a gravity of 2.40, thus making it appear to be a satisfactory pebble when it was not. The most that such a condition could show would be to indicate that the unsaturated pebble was somewhat more unsatisfactory than it actually was but since it does not pass the flotation test anyway its degree of unsuitability is irrelevant.

DESIGNING CONCRETE MIXTURES FOR PAVEMENTS

BY W. F. KELLERMANN[1]

SYNOPSIS

The purpose of this paper is to describe a method of investigating the flexural strength of concrete in connection with the problem of designing concrete mixtures for pavements, and to present the results of a series of laboratory tests which demonstrate how flexural strength may vary over a wide range due to the characteristics of the aggregates employed.

It will be taken for granted at the outset that all pavement concrete, particularly that laid in the northern states, must be designed so as to afford maximum resistance to weathering agencies. Assuming that the constituent materials are durable, it is generally agreed that when normally graded aggregates are used this may be accomplished either by placing a maximum limit on the water-cement ratio or by requiring a cement content sufficiently high to insure that the maximum allowable water content will not be exceeded. The necessity for limiting the water-cement ratio to insure durability applies to all concrete exposed to the weather. In so far as strength characteristics are concerned, concrete for most purposes need only be investigated for compressive strength. However, compressive strength is not of primary importance in concrete for pavement slabs because of the character of the stresses to which such slabs are subjected. Live loads and changes in temperature and moisture, either alone or in combination, produce tensile and flexural stresses which pavements must resist in order to perform the function for which they are designed. Of the two, the flexural stresses are the more important. For this reason, flexural or bending stresses rather than compressive stresses become critical. Therefore, it becomes the problem of the designer of concrete paving mixtures to give consideration not only to the factors which affect durability but also to those variables which affect flexural strength.

The specifications for pavement concrete of the American Association of State Highway Officials specify that the proportions shall be based on laboratory tests and shall be such that, in the judgment of the engineer, they will assure durable concrete of the plasticity and workability required, and which will attain at the age of 14 days a modulus of rupture not less than 550 psi. when tested by the third-point method of loading. In order to assure durability it is further specified that the net water-cement ratio shall in no case exceed 6.0 gal. per sack of cement.

The tests reported in this paper were obtained in an investigation of the design of concrete mixtures in which

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25 different combinations of fine and coarse aggregate were used, the requirement being compliance with the above specification. The work was done in the laboratory of the Public Roads Administration during 1936 at the request of the State Highway and Public Works Commission of North Carolina. The purpose was to establish the cement factor required for various combinations of available aggregates, the information thus obtained to be used as the basis for bidding. Seven sands and 13 coarse aggregates, all commercially available in North Carolina, were investigated. The 25 combinations of materials were selected on the basis of economic availability and represented practically all combinations of aggregates that were likely to be encountered in practice in that state.

As will be noted from Table I, the sands varied in grading over a wide range, the fineness modulus of the finest being 2.12 and that of the coarsest, 3.37. All coarse aggregates were separated into three sizes and recombined for test in accordance with the grading shown in Table II, the maximum size being

TABLE I.—SIEVE ANALYSIS OF FINE AGGREGATES.

Fine Aggregate	Percentage Retained on						Fineness Modulus
	Sieve No. 4	Sieve No. 8	Sieve No. 16	Sieve No. 30	Sieve No. 50	Sieve No. 100	
No. 1....	0	0	2.8	35.8	77.3	95.9	2.12
No. 2....	0	0	5.3	42.8	79.4	94.4	2.22
No. 3....	0.5	4.0	19.6	58.0	93.1	99.4	2.75
No. 4....	0.2	4.1	21.2	63.2	93.2	99.0	2.81
No. 5....	0	7.3	26.8	62.1	89.3	97.2	2.83
No. 6....	0.1	7.1	30.4	68.3	91.0	98.4	2.95
No. 7....	0.3	14.6	44.2	81.2	97.6	99.4	3.37

TABLE II.—PHYSICAL PROPERTIES OF AGGREGATES.

Aggregate	Type	Bulk Specific Gravity	Weight, lb. per cu. ft.[a]		Voids, per cent		Absorption, per cent	Abrasion Loss, per cent	
			Dry Rodded	Dry Loose	Dry Rodded	Dry Loose		Los Angeles[b]	Deval[c]
				Stone					
No. 1.......	Dolomite	2.85	105	97	41	45	0.27	30.4	4.3
No. 2.......	Dolomite	2.82	103	93	42	47	0.56	33.0	5.0
No. 3.......	Limestone	2.79	102	92	41	47	0.44	21.1	2.6
No. 4.......	Granite	2.69	101	91	40	46	0.46	51.1	4.0
No. 5.......	Granite	2.65	102	93	38	44	0.46	53.5	2.6
No. 6.......	Granite	2.64	102	92	38	44	0.58	37.9	2.4
No 7.......	Granite	2.63	100	91	39	44	0.52	58.4	3.9
No. 8.......	Granite	2.62	95	87	42	47	0.28	30.1	2.0
No. 9.......	Granite	2.62	100	90	39	45	0.44	41.9	3.6
				Gravel					
No. 10	Gneiss	2.67	106	98	36	41	1.19	47.9	18.5
No. 11......	Quartz	2.63	111	103	32	37	0.22	53.7	14.6
No. 12......	Quartz	2.63	109	103	34	37	0.28	40.9	8.2
No. 13......	Quartz	2.63	111	104	32	37	0.32	54.9	19.4
				Sand					
No. 1.......	Quartz	2.64	98	91	40	45	0.40		
No. 2.......	Quartz	2.63	100	93	39	43	0.50		
No. 3.......	Quartz	2.63	98	93	40	43	0.40		
No. 4.......	Quartz	2.64	98	92	40	44	0.40		
No. 5.......	Quartz	2.66	103	97	38	42	0.35		
No. 6.......	Quartz	2.64	101	94	39	43	0.32		
No. 7.......	Quartz	2.60	99	94	39	42	0.70		

[a] All coarse aggregates proportioned to give following grading in concrete. Unit weights determined for this grading:

Total retained on 2-in. sieve	0 per cent
Total retained on 1½-in. sieve	15 per cent
Total retained on ¾-in. sieve	70 per cent
Total retained on No. 4 sieve	100 per cent
Fineness modulus	7.75

[b] Grading A used with both stone and gravel.

[c] Grading A used with gravel. Stone sample consisted of 50 pieces weighing 5 kg.

2 in. This table also gives the mineral composition of both the fine and coarse aggregates as well as their physical properties. One lot of cement, meeting all A.S.T.M. requirements, was used throughout.

PROPORTIONING

In order to determine the required cement content for each combination of materials, it was decided to establish directly the relationship between cement content and flexural strength at 14 days, using mixtures with five different cement factors as follows: 4.4, 5.2, 6.0, 6.8, and 7.2 sacks of cement per cubic yard of concrete. This procedure also afforded an opportunity to establish the corresponding relationship between water-cement ratio and flexural strength.

The decision to design the mixes on the basis of a fixed cement factor rather than by the use of fixed water-cement ratios was made because of the fact that in the North Carolina specifications the final proportions are stated in terms of a fixed cement factor for each aggregate combination. The problem therefore resolved itself into one of designing 125 different concrete mixes; five cement contents with each of the 25 combinations of aggregates. The problem was complicated by the fact that both angular and rounded coarse aggregates were used in combination with sands graded from extremely fine to extremely coarse. In keeping with North Carolina practice the different mixes were designed with a view to maintaining a minimum of sand consistent with satisfactory workability at a consistency corresponding to a slump of $2\frac{1}{2}$ in. This was accomplished by making numerous trial batches, the ratio of fine to coarse aggregate being adjusted in each case until, in the opinion of the operator, the minimum sand content was reached.

The final proportions for each of the five cement factors and for each of the 25 aggregate combinations are shown in Table III. This table includes, in addition to the mix proportions by weight, the water-cement ratio by volume, the value of W_c, that is, the volume of water in a unit volume of concrete, the ratio b/b_o as defined by Talbot and Richart,[2] the mortar voids ratio,[3] the percentage of sand by weight, the fineness modulus of the combined aggregate and the resulting slump in inches.

In view of the fact that it was necessary to design 25 different mixes for each cement content, attempts were made to apply certain theories of mix design to the problem of determining the proper percentage of sand to use in each case. An attempt to use the fineness modulus theory of Abrams[4] proved unsuccessful due to the fact that a fixed value for maximum permissible fineness modulus could not be used, even in the case of a given sand combined with several coarse aggregates of the same general particle shape. Next, an attempt was made to design on the basis of a fixed value for the mortar voids ratio (that is, a constant excess of mortar, expressed on a percentage basis, over the amount necessary to fill the voids in the coarse aggregate). This was found satisfactory as long as particle shape remained reasonably constant. However, as will be noted in Table III, a change from angular to rounded coarse aggregate resulted in an increase in the value of this ratio.

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[2] A. N. Talbot and F. E. Richart, "The Strength of Concrete—Its Relation to the Cement, Aggregate and Water," *Bulletin 137*, Engineering Experiment Station, University of Illinois, October 15, 1923. The term b/b_0 is defined as the ratio of the absolute volume of coarse aggregate in a unit volume of concrete (b) to the absolute volume of coarse aggregate in a unit volume of coarse aggregate (b_0). Stated in different terms, it is the apparent volume of coarse aggregate in a unit volume of concrete. The values given in Table III are on a dry-loose basis.

[3] The ratio of the volume of mortar in a unit volume of concrete to the volume of the voids in the coarse aggregate determined in a dry-loose condition.

[4] D. A. Abrams, "The Design of Concrete Mixtures," *Bulletin No. 1*, Structural Materials Research Laboratory, Lewis Institute (1919).

TABLE III.—DATA ON MIXES.

Aggregate		Proportions by Weight, lb.	Water-Cement Ratio, by volume	W_c*	b/b_o**	Mortar Voids Ratio	Sand to Total Aggregate, by weight, per cent	Combined Fineness Modulus	Slump, in.
Fine	Coarse								
Cement Factor—4.4 sacks per cubic yard.									
No. 1	No. 1	94:254:548	1.04	0.169	0.920	1.19	31.7	5.88	2.4
	No. 2	94:264:524	1.08	0.176	0.920	1.18	33.5	5.79	2.3
	No. 3	94:267:519	1.06	0.173	0.920	1.18	34.0	5.77	2.3
	No. 10	94:226:553	0.97	0.158	0.920	1.21	29.0	6.11	2.5
No. 2	No. 4	94:256:514	1.04	0.169	0.920	1.19	33.2	5.88	2.5
	No. 5	94:240:526	1.02	0.166	0.920	1.20	31.3	6.01	2.7
	No. 6	94:245:520	1.01	0.164	0.920	1.20	32.0	5.98	2.5
	No. 7	94:246:515	1.02	0.166	0.920	1.20	32.3	5.96	2.4
	No. 8	94:263:492	1.05	0.171	0.920	1.19	34.8	5.83	2.6
	No. 9	94:251:508	1.02	0.166	0.920	1.19	33.1	5.93	2.4
	No. 11	94:205:583	0.86	0.140	0.920	1.23	26.0	6.31	2.6
	No. 13	94:200:587	0.86	0.140	0.920	1.24	25.4	6.35	2.5
No. 3	No. 4	94:272:492	1.07	0.174	0.880	1.30	35.6	5.94	2.4
	No. 5	94:263:501	1.03	0.168	0.880	1.31	34.4	6.02	2.6
	No. 6	94:263:497	1.04	0.169	0.880	1.31	34.6	6.02	2.5
	No. 7	94:267:492	1.03	0.168	0.880	1.31	35.2	5.99	2.4
	No. 9	94:272:486	1.03	0.168	0.880	1.30	35.9	5.96	2.5
No. 4	No. 13	94:224:550	0.95	0.155	0.860	1.45	28.9	6.32	2.6
No. 5	No. 7	94:274:492	1.01	0.164	0.880	1.31	35.8	6.00	2.5
	No. 9	94:276:486	1.03	0.168	0.880	1.30	36.2	5.98	2.5
No. 6	No. 4	94:280:480	1.10	0.179	0.860	1.36	36.8	5.96	2.4
	No. 6	94:277:486	1.03	0.168	0.860	1.37	36.3	6.01	2.4
	No. 11	94:237:545	0.90	0.147	0.860	1.44	30.3	6.30	2.6
No. 7	No. 7	94:297:458	1.04	0.169	0.820	1.50	39.3	6.02	2.3
	No. 12	94:261:519	0.89	0.145	0.820	1.59	33.5	6.27	2.4
Cement Factor—5.2 sacks per cubic yard.									
No. 1	No. 1	94:208:459	0.87	0.168	0.910	1.22	31.2	5.90	2.5
	No. 2	94:216:438	0.91	0.175	0 910	1.21	33.0	5.81	2.6
	No. 3	94:217:434	0.90	0.173	0.910	1.21	33.3	5.80	2.5
	No. 10	94:183:463	0.82	0.158	0.910	1.24	28.3	6.15	2.6
No. 2	No. 4	94:207:430	0.89	0.171	0.910	1.22	32.5	5.93	2.6
	No. 5	94:199:438	0.85	0.164	0.910	1.23	31.2	6.02	2.6
	No. 6	94:199:435	0.86	0.166	0.910	1.23	31.4	6.01	2.6
	No. 7	94:202:430	0.86	0.166	0.910	1.22	32.0	5.99	2.5
	No. 8	94:215:410	0.89	0.171	0.910	1.21	34.4	5.85	2.5
	No. 9	94:205:425	0.86	0.166	0.910	1.22	32.5	5.95	2.6
	No. 11	94:169:486	0.72	0.139	0.910	1.27	25.8	6.32	2.5
	No. 13	94:162:491	0.73	0.141	0.910	1.27	24.8	6.37	2.5
No. 3	No. 4	94:225:411	0.89	0.171	0.870	1.32	35.4	5.96	2.5
	No. 5	94:217:420	0.85	0.164	0.870	1.34	34.1	6.04	2.6
	No. 6	94:218:417	0.85	0.164	0.870	1.34	34.3	6.02	2.6
	No. 7	94:218:412	0.87	0.168	0.870	1.33	34.6	6.02	2.5
	No. 9	94:222:407	0.87	0.168	0.870	1.33	35.3	5.99	2.6
No. 4	No. 13	94:184:459	0.79	0.152	0.850	1.48	28.6	6.34	2.6
No. 5	No. 7	94:224:412	0.85	0.164	0.870	1.33	35.2	6.03	2.6
	No. 9	94:224:407	0.87	0.168	0.870	1.33	35.5	6.02	2.6
No. 6	No. 4	94:231:401	0.92	0.177	0.850	1.39	36.6	5.98	2.7
	No. 6	94:224:407	0.88	0.170	0.850	1.40	35.5	6.05	2.6
	No. 11	94:194:455	0.76	0.146	0.850	1.48	29.9	6.31	2.6
No. 7	No. 7	94:243:382	0.88	0.170	0.810	1.53	38.9	6.03	2.5
	No. 12	94:212:433	0.76	0.146	0.810	1.63	32.9	6.30	2.5
Cement Factor—6.0 sacks per cubic yard.									
No. 1	No. 1	94:173:393	0.76	0.169	0.900	1.24	30.6	5.94	2.5
	No. 2	94:181:377	0.78	0.173	0.900	1.24	32.4	5.84	2.6
	No. 3	94:181:373	0.78	0.173	0.900	1.24	32.7	5.84	2.7
	No. 10	94:153:397	0.71	0.158	0.900	1.27	27.8	6.17	2.6
No. 2	No. 4	94:174:369	0.76	0.169	0.900	1.24	32.0	5.95	2.5
	No. 5	94:162:377	0.75	0.167	0.900	1.25	30.1	6.07	2.6
	No. 6	94:166:372	0.75	0.167	0.900	1.25	30.9	6.04	2.5
	No. 7	94:167:369	0.75	0.167	0.900	1.25	31.2	6.02	2.6
	No. 8	94:181:352	0.77	0.171	0.900	1.24	34.0	5.88	2.5
	No. 9	94:167:365	0.77	0.171	0.900	1.25	31.4	6.01	2.6
	No. 11	94:138:417	0.64	0.142	0.900	1.30	24.9	6.37	2.5
	No. 13	94:133:422	0.64	0.142	0.900	1.30	24.0	6.42	2.4

* Volume of water per unit volume of concrete.

** See footnote 2 in text.

TABLE III.—DATA ON MIXES—*Continued.*

Aggregate		Proportions by Weight, lb.	Water-Cement Ratio, by volume	W_c*	b/b_o**	Mortar Voids Ratio	Sand to Total Aggregate, by weight, per cent	Combined Fineness Modulus	Slump, in.
Fine	Coarse								
Cement Factor—6.0 sacks per cubic yard—*Continued.*									
No. 3	No. 4	94:189:353	0.77	0.171	0.860	1.36	34.9	5.98	2.7
	No. 5	94:182:359	0.74	0.164	0.860	1.37	33.6	6.06	2.7
	No. 6	94:184:356	0.74	0.164	0.860	1.37	34.1	6.04	2.6
	No. 7	94:185:353	0.74	0.164	0.860	1.37	34.4	6.02	2.4
	No. 9	94:187:348	0.75	0.167	0.860	1.36	35.0	6.00	2.7
No. 4	No. 13	94:155:394	0.68	0.151	0.840	1.52	28.2	6.36	2.5
No. 5	No. 7	94:189:353	0.73	0.162	0.860	1.37	34.9	6.04	2.6
	No. 9	94:191:348	0.74	0.164	0.860	1.36	35.4	6.02	2.6
No. 6	No. 4	94:196:344	0.78	0.173	0.840	1.42	36.3	5.99	2.5
	No. 6	94:191:348	0.75	0.167	0.840	1.43	35.4	6.05	2.4
	No. 11	94:165:389	0.65	0.144	0.840	1.51	29.8	6.32	2.3
No. 7	No. 7	94:206:328	0.75	0.167	0.800	1.56	38.6	6.05	2.4
	No. 12	94:178:371	0.66	0.147	0.800	1.67	32.4	6.32	2.5
Cement Factor—6.8 sacks per cubic yard.									
No. 1	No. 1	94:150:343	0.65	0.164	0.890	1.28	30.4	5.95	2.3
	No. 2	94:156:327	0.68	0.171	0.890	1.26	32.3	5.85	2.6
	No. 3	94:158:324	0.67	0.169	0.890	1.26	32.8	5.84	2.6
	No. 10	94:128:347	0.63	0.159	0.890	1.30	26.9	6.21	2.6
No. 2	No. 4	94:148:321	0.68	0.171	0.890	1.27	31.6	5.98	2.5
	No. 5	94:139:327	0.66	0.166	0.890	1.28	29.8	6.09	2.6
	No. 6	94:143:324	0.65	0.164	0.890	1.28	30.6	6.06	2.5
	No. 7	94:141:322	0.67	0.169	0.890	1.28	30.5	6.06	2.6
	No. 8	94:153:307	0.68	0.171	0.890	1.27	33.3	5.92	2.6
	No. 9	94:144:317	0.67	0.169	0.890	1.28	31.2	6.02	2.6
	No. 11	94:115:364	0.57	0.144	0.890	1.34	24.0	6.42	2.5
	No. 13	94:110:368	0.58	0.146	0.890	1.34	23.0	6.48	2.5
No. 3	No. 4	94:164:307	0.66	0.166	0.850	1.39	34.8	5.98	2.5
	No. 5	94:156:313	0.65	0.164	0.850	1.41	33.3	6.08	2.6
	No. 6	94:159:310	0.64	0.161	0.850	1.40	33.9	6.05	2.5
	No. 7	94:159:307	0.65	0.164	0.850	1.40	34.1	6.04	2.6
	No. 9	94:161:302	0.66	0.166	0.850	1.39	34.8	6.02	2.6
No. 4	No. 13	94:130:343	0.61	0.154	0.830	1.55	27.5	6.40	2.6
No. 5	No. 7	94:161:307	0.65	0.164	0.850	1.40	34.4	6.07	2.6
	No. 9	94:164:302	0.65	0.164	0.850	1.39	35.2	6.03	2.7
No. 6	No. 4	94:165:301	0.70	0.176	0.830	1.44	35.4	6.03	2.5
	No. 6	94:161:303	0.67	0.169	0.830	1.46	34.7	6.08	2.6
	No. 11	94:137:340	0.59	0.149	0.830	1.54	28.7	6.38	2.5
No. 7	No. 7	94:178:286	0.65	0.164	0.790	1.59	38.4	6.05	2.5
	No. 12	94:152:323	0.58	0.146	0.790	1.71	32.0	6.34	2.4
Cement Factor—7.2 sacks per cubic yard.									
No. 1	No. 1	94:137:322	0.63	0.168	0.885	1.28	29.8	5.98	2.6
	No. 2	94:143:308	0.65	0.173	0.885	1.27	31.7	5.88	2.6
	No. 3	94:145:305	0.64	0.171	0.885	1.27	32.2	5.86	2.5
	No. 10	94:119:325	0.60	0.160	0.885	1.31	26.8	6.23	2.6
No. 2	No. 4	94:136:302	0.64	0.171	0.885	1.28	31.1	6.00	2.5
	No. 5	94:128:309	0.62	0.165	0.885	1.30	29.3	6.12	2.5
	No. 6	94:130:306	0.62	0.165	0.885	1.29	29.8	6.10	2.5
	No. 7	94:133:302	0.62	0.165	0.885	1.29	30.6	6.06	2.4
	No. 8	94:141:289	0.64	0.171	0.885	1.28	32.8	5.94	2.4
	No. 9	94:135:299	0.62	0.165	0.885	1.29	31.1	6.04	2.4
	No. 11	94:105:341	0.55	0.147	0.885	1.35	23.5	6.45	2.5
	No. 13	94:102:345	0.55	0.147	0.885	1.35	22.8	6.49	2.4
No. 3	No. 4	94:151:289	0.63	0.168	0.845	1.40	34.3	6.01	2.6
	No. 5	94:144:294	0.61	0.163	0.845	1.42	32.9	6.10	2.6
	No. 6	94:146:292	0.61	0.163	0.845	1.41	33.3	6.08	2.6
	No. 7	94:146:289	0.62	0.165	0.845	1.41	33.6	6.07	2.6
	No. 9	94:149:284	0.62	0.165	0.845	1.41	34.4	6.04	2.6
No. 4	No. 13	94:120:322	0.58	0.155	0.825	1.58	27.1	6.41	2.5
No. 5	No. 7	94:149:289	0.61	0.163	0.845	1.41	34.0	6.09	2.6
	No. 9	94:151:285	0.62	0.165	0.845	1.41	34.6	6.06	2.5
No. 6	No. 4	94:155:280	0.66	0.176	0.825	1.46	35.6	6.02	2.5
	No. 6	94:150:285	0.63	0.168	0.825	1.49	34.5	6.09	2.4
	No. 11	94:127:318	0.56	0.149	0.825	1.57	28.5	6.39	2.5
No. 7	No. 7	94:165:267	0.62	0.165	0.785	1.62	38.2	6.06	2.5
	No. 12	94:141:304	0.55	0.147	0.785	1.74	31.7	6.35	2.5

(Compare combinations 2–4 to 2–9, inclusive, with combinations 2–11 and 2–13.)

This particular difficulty was overcome by using a fixed value of b/b_o in place of a constant mortar-voids ratio. After making many trial batches it was found that, for a given cement content and a given sand, a constant value of b/b_o could be used irrespective of type of coarse aggregate. (See Table III.) It was noted, furthermore, that certain consistent changes in b/b_o resulted from changing the cement factor and sand grading. Thus, for a given sand, Table III shows that an increase in the cement factor of 0.8 sack resulted in a decrease of 0.01 in b/b_o for equal workability. Furthermore, as the sand became coarser, it was found necessary to reduce the value of b/b_o. Thus at 6.0 sacks per cubic yard, the value of this ratio decreased from 0.90 for sand No. 1 (fineness modulus = 2.12) to 0.80 for sand No. 7 (fineness modulus = 3.37).

It is believed that the systematic variation in b/b_o with changes in cement content revealed by these tests together with the principle stated by Lyse[5] in 1932 to the effect that, for a given combination of materials and a given consistency the total quantity of water per unit of volume of concrete (W_c) is constant regardless of the cement content, makes it possible to simplify considerably the problem of designing mixtures of varying cement content. Having established by trial the proper value of b/b_o to use with given aggregates and with a given cement factor, the proportions required for the same consistency with any other cement factor may be obtained by computation, provided the aggregates have the same gradations as those used in the trial batch. This is illustrated by the following example:

Given a mix having the proportions
94:274:492 by weight,
water-cement ratio 1.01 by volume,
Specific gravities of materials:
cement, 3.16; fine aggregate, 2.66; coarse aggregate, 2.63

Percentage of voids in coarse aggregate (dry loose), 44.5, the corresponding solid volumes per 1-sack batch would be as follows:

Cement...... $\dfrac{94}{62.4 \times 3.16} = 0.48$ cu. ft.

Fine aggregate..... $\dfrac{274}{62.4 \times 2.66} = 1.65$ cu. ft.

Coarse aggregate..... $\dfrac{492}{62.4 \times 2.63} = 3.00$ cu. ft.

Water (w/c)............... = $\underline{1.01}$ cu. ft.

Yield = 6.14 cu. ft.

$$\text{Cement factor} = \frac{27.0}{6.14} = 4.4 \text{ sacks per cubic yard}$$

Bulk volume of coarse aggregate =

$$\frac{3.00}{1.00} - 0.445 = 5.41 \text{ cu. ft.}$$

$$\text{Then } b/b_o = \frac{5.41}{6.14} = 0.88$$

Water per unit volume of concrete (W_c) =

$$\frac{1.01}{6.14} = 0.164$$

Having analyzed the above mix and determined the values of b/b_o and W_c as 0.88 and 0.164, respectively, let us now design another mix with the same materials but with a different cement factor, say 6.8 sacks of cement per cubic yard of concrete, an increase of 2.4 sacks per cubic yard.

Following the procedure outlined, the value for W_c for the new mix would remain constant at 0.164. The new value for b/b_o is determined as follows: For each increase of 0.8 sack per cubic yard in cement factor, b/b_o is decreased 0.01. Therefore, the new value of b/b_o is $0.88 - \dfrac{2.4}{0.8} \times 0.01 = 0.85$.

The yield per 1-sack batch for the new mix would be $\dfrac{27}{6.8} = 3.97$ cu. ft. and the

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[5] Inge Lyse, "Tests of Consistency and Strength of Concrete Having Constant Water Content," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 629 (1932).

apparent or bulk volume of coarse aggregate would be 3.97 × 0.85 = 3.37 cu. ft. The corresponding solid volume of coarse aggregate (b)..$3.37 \times (1 - 0.445) = 1.87$ cu. ft.
The solid volume of cement (c)...0.48 cu. ft.
The volume of water (w/c)..$3.97 \times 0.164 = 0.65$ cu. ft.
Total......................3.00 cu. ft.

The only unknown quantity remaining is the fine aggregate, which is obtained by subtracting the sum of the solid volumes of coarse aggregate, cement, and water from the total yield. Therefore the solid volume of fine aggregate = 3.97 − 3.00 = 0.97 cu. ft. The complete proportions per 1-sack batch for the new mix would be as follows:

Cement	0.48 cu. ft. solid
Fine aggregate	0.97 cu. ft. solid
Coarse aggregate	1.87 cu. ft. solid
Water (w/c)	0.65 cu. ft.
Total	3.97 cu. ft. solid

Multiplying the above by 62.4 times the appropriate values for specific gravity gives the following weight proportions:

94:161:307
$w/c = 0.65$ (by volume)

By this procedure any mix, within a reasonable range, can readily be calculated provided the proper value of b/b_o and W_c have been predetermined on the materials under investigation and, further, provided the slump is to remain constant. As can be seen from the example given above, the only unknown quantity in the mix is the sand and this is arrived at by simple calculation. In these tests the water-cement ratio arrived at by calculation from the law of constant water per unit volume of concrete for a constant slump was not always the exact value needed to obtain the proper slump. However, it was possible in all cases to make the proper adjustment by slight changes in the ratio of water to sand, keeping the sum of the absolute volumes of the two ingredients constant. Even though the general law did not hold precisely in all individual cases, that is, to the third decimal, the following table, which gives average values for the 25 combinations, will illustrate its accuracy:

Cement Factor, sacks per cu. yd.	W_c
4.4	0.164
5.2	0.163
6.0	0.163
6.8	0.163
7.2	0.163

In connection with the procedure employed, that is, the use of the highest value of b/b_o compatible with workability, it is interesting to note that for a given coarse aggregate and a given cement factor it was possible to use approximately the same water-cement ratio irrespective of whether a coarse or fine sand was used. For instance, in Table III, cement factor 6.0 sacks per cubic yard, coarse aggregate No. 7 was used with sands Nos. 2, 3, 5, and 7. These sands varied in fineness modulus

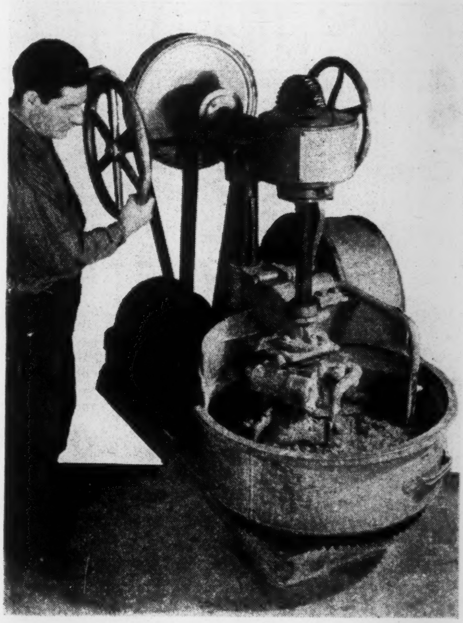


FIG. 1.—Laboratory Mixer Used in Preparing Test Mixtures.

from 2.22 to 3.37. However, the total range in water-cement ratio was only 0.02 (from 0.73 to 0.75), demonstrating that, by proper proportioning, it is possible to maintain a low water-cement ratio when using fine sands.

TABLE IV.—AVERAGE FLEXURAL STRENGTH, 14 DAYS.

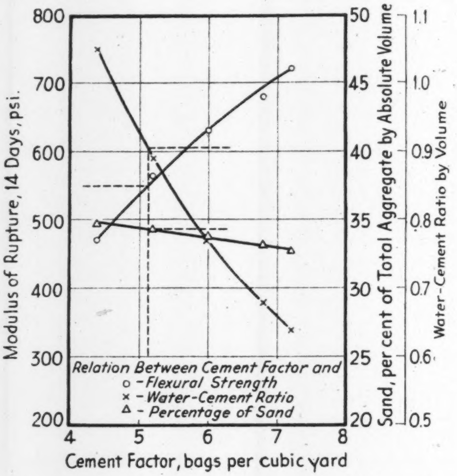
6 by 6-in. beams tested on 18-in. span with third-point loading.

Each of the values is an average of five tests.

Aggregate		Modulus of Rupture, psi.				
Fine	Coarse	Cement Factor, 4.4 sacks per cu. yd.	Cement Factor, 5.2 sacks per cu. yd.	Cement Factor, 6.0 sacks per cu. yd.	Cement Factor, 6.8 sacks per cu. yd.	Cement Factor, 7.2 sacks per cu. yd.
No. 1	No. 1	470	615	655	795	765
	No. 2	460	575	655	695	755
	No. 3	435	570	715	730	745
	No. 10	380	425	510	535	545
No. 2	No. 4	455	510	540	630	655
	No. 5	430	505	575	635	630
	No. 6	475	570	620	640	615
	No. 7	440	505	555	610	640
	No. 8	470	565	630	680	720
	No. 9	470	565	600	665	680
	No. 11	490	500	545	635	635
	No. 13	430	485	535	560	615
No. 3	No. 4	350	470	570	600	610
	No. 5	340	470	560	600	630
	No. 6	355	470	565	645	630
	No. 7	365	445	575	610	625
	No. 9	390	495	620	665	660
No. 4	No. 13	430	485	510	585	585
No. 5	No. 7	395	490	565	610	670
	No. 9	395	510	615	675	695
No. 6	No. 4	425	490	530	630	620
	No. 6	465	545	585	685	670
	No. 11	480	515	575	635	655
No. 7	No. 7	395	490	565	630	620
	No. 12	505	555	630	675	685
Average		428	513	584	642	654

FABRICATING SPECIMENS

The concrete was mixed in a laboratory mixer of the type shown in Fig. 1. In order to approximate field conditions, the coarse aggregate was handled in a saturated surface-dry condition and the sand in a wet condition, correction for the free water in the sand being made when computing the water-cement ratio. Test specimens consisted of 6 by 6-in. beams, 21 in. long. In all, 625 specimens—25 combinations of material, times 5 mixes, times 5 specimens per mix made on each of 5 different working days—were fabricated. All tests were made at 14 days, the specimens being tested by the third-point method in accordance with A.S.T.M. Standard Methods of Test for Flexural Strength of Concrete (C 78 – 39).[6] The specimens were placed in the testing machine with the side, as molded, in tension.



Aggregate	Number	Type	Specific Gravity	Absorption, per cent	Fineness Modulus
Fine	2	Sand	2.63	0.50	2.22
Coarse	8	Granite	2.62	0.28	7.75

FIG. 2.—North Carolina Mix Design Chart for Combination 2–8.

DISCUSSION OF RESULTS

The average flexural strengths for each combination of materials for each of the five different proportions are given in Table IV. In discussing these results the various combinations of materials will be referred to by number. Thus, combination 2–4 refers to sand

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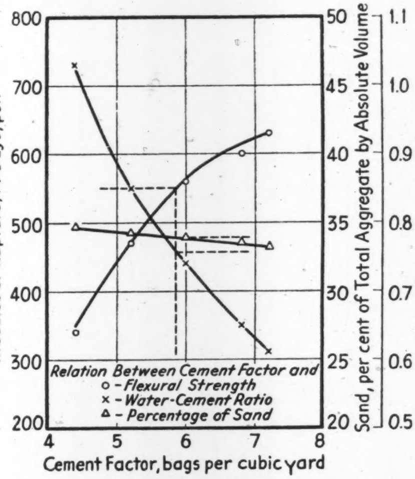
[6] 1939 Book of A.S.T.M. Standards, Part II, p. 339.

No. 2 combined with coarse aggregate No. 4. In order to illustrate the method of plotting the results for the purpose of determining the required cement factor for a specified modulus of rupture, two examples will be given.

Figure 2 shows a typical chart giving the relationship between modulus of rupture and cement factor, water-cement ratio and cement factor, and percentage of sand and cement factor. Considering first the strength-cement factor relationship, it will be noted from the curve that the required amount of cement to produce a modulus of rupture of 550 psi. was 5.1 sacks per cubic yard. The corresponding water-cement ratio was 0.91 and the percentage of sand 34. From this type of chart a complete mix design could be computed for any strength specified within the range covered. In Fig. 2 for instance, the range in strength would be from about 500 to 700 psi.



Aggregate	*Number*	*Type*	*Specific Gravity*	*Absorption, per cent*	*Fineness Modulus*
Fine	*3*	*Sand*	*2.63*	*0.40*	*2.75*
Coarse	*5*	*Granite*	*2.65*	*0.46*	*7.75*

FIG. 3.—North Carolina Mix Design Chart for Combination 3–5.

Frequently specifications for concrete contain a limiting value for water-cement ratio in order to assure durability. Assuming a maximum allowable value of 6 gal. per sack (water-cement ratio of 0.80 by volume), it will be observed from Fig. 2 that the required strength was obtained with a water-cement ratio of 0.91, which is more than allowable. From the water-cement ratio – cement factor curve it is seen that, in order to keep within the limits dictated by durability considerations, it would be necessary to use a cement factor of 5.8 sacks per cubic yard instead of 5.1 sacks, the resulting flexural strength being in excess of 600 psi.

TABLE V.—CEMENT FACTOR REQUIRED FOR 550 PSI. MODULUS OF RUPTURE.
Third-point loading.

Fine Aggregate	Coarse Aggregate	Cement Factor, sacks per cu. yd.
No. 1	No. 1	4.9
	No. 2	5.1
	No. 3	5.1
	No. 10	7.1
No. 2	No. 4	5.8
	No. 5	5.7
	No. 6	5.1
	No. 7	6.0
	No. 8	5.1
	No. 9	5.0
	No. 11	5.8
	No. 13	6.2
No. 3	No. 4	5.8
	No. 5	5.9
	No. 6	5.9
	No. 7	6.1
	No. 9	5.5
No. 4	No. 13	6.4
No. 5	No. 7	5.9
	No. 9	5.5
No. 6	No. 4	6.0
	No. 6	5.3
	No. 11	5.6
No. 7	No. 7	5.9
	No. 12	5.0

Figure 3 gives the same type of data for a different combination of materials. The required cement factor as determined from the curve is 5.9 sacks, with a corresponding water-cement ratio of 0.75. In this case the strength is the governing factor while in the former case the maximum allowable water-cement ratio governs.

Charts similar to Figs. 2 and 3 were drawn for each of the 25 combinations of materials and the required cement factors obtained from the strength curves as illustrated. These cement factors are enumerated in Table V and show values ranging from 4.9 sacks for combination 1–1 to 7.1 sacks for combination 1–10, or, for all practical purposes, from 5 to 7 sacks. In view of the fact that the same sand was used in both combinations, the strength differential in this case is a direct function of the concrete-making properties of the two coarse aggregates.

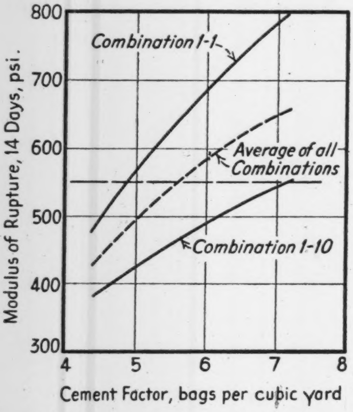


FIG. 4.—Relation Between Cement Factor and Flexural Strength of Concrete. Average curve and maximum range.

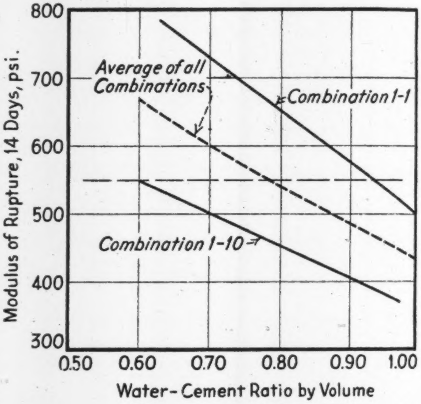


FIG. 5.—Relation Between Water-Cement Ratio and Flexural Strength of Concrete. Average curve and maximum range.

In order better to illustrate the effect of aggregate upon flexural strength, Fig. 4 is shown. Three flexural strength–cement factor curves are given, one for combination 1–1, one for combination 1–10 and one for the average of all 25 combinations of materials, thus representing the average and the extreme ranges for the entire series of tests. From the shapes of the two extreme curves it is seen that they tend to converge at the lower cement factors. This is to be expected because of the fact that for very lean mixes the flexural strength of the concrete is practically the strength of the mortar. However, as the cement content is increased and the mortar becomes stronger, the quality of the coarse aggregate and the bond between mortar and coarse aggregate become important factors. In the case of coarse aggregate No. 1, we have an excellent quality of dolomite having a surface texture which gives good bond with the mortar. Under these conditions, the full strength of the mortar appeared to have been developed up to a cement factor of 7 sacks per cubic yard. In the case of aggregate No. 10, we have a structurally weaker material so that the full strength of the mortar was not developed. If the water-cement ratio was the only controlling factor, the strengths of combination 1–10 should have been greater than those developed by combination 1–1 because of the fact that lower water-cement ratios were used (Table III). Notwithstanding this fact, combination 1–1 gave 26 per cent greater strength for the leanest mix and 44 per cent greater strength for the richest mix. At a cement factor of 6 sacks per cubic yard, one commonly used for pavement work, combination 1–1 gave 40 per cent greater strength than combination 1–10. While in this particular instance the low strength is probably due to structurally poor material, this is not always the case. Frequently aggregates that are structurally strong give comparatively low flexural strengths in concrete because of the fact that the surface texture does not permit of sufficient bond to develop the full strength of the mortar.

Figure 5 shows the strength data given in Fig. 4 plotted against the water-cement ratio instead of the cement factor. Here again the quality of the coarse aggregate is reflected in the results obtained. Note, for instance, that a water-cement ratio of 0.6 was required to develop a modulus of rupture of 550 psi. with combination 1–10, whereas this strength was obtained with a water-cement ratio of 0.95 in the case of combination 1–1.

Attention is directed to the fact that the curves for combinations 1–1 and 1–10 are straight lines. However, this was not the case for all combinations of materials investigated, many combinations giving curves which had the characteristic shape of the typical water-cement ratio – compressive strength curve. In fact the average curve shown is of this general shape.

The data shown in Figs. 2 and 3 might have been plotted with the principal relationship between water-cement ratio and strength as in Fig. 5, with additional curves to give the other desired information. The arrangement of data is of course a matter to be decided by circumstances.

Complete compressive strength data are not available for this series of tests because of the fact that companion compressive strength specimens were not made. However, limited data were obtained for the three leanest mixes (4.4, 5.2, and 6.0 sacks of cement per cubic yard) as the result of tests on modified cubes remaining from the flexure tests. From these data the average results for combination 1–1 were found to be 9 per cent greater in compressive strength than those for combination 1–10 as compared with the corresponding average flexural strength differential of 32 per cent. Another case is that of the group in which sand No. 2 was combined with eight different coarse aggregates. Here combination 2–13 gave just about as high compressive strength as any combination in the group. However, it was found that, at a cement factor of 6 sacks per cubic yard, the flexural strength of combination 2–13 was the lowest of all, other combinations in the group giving strengths up to 17 per cent higher than combination 2–13 (Table IV). Many other examples could be given to show that two concretes having the same compressive strength do not necessarily have the same flexural strength, emphasizing the importance of determining the flexural strength characteristics of constituent materials used in pavement slabs.

CONCLUSIONS

It is believed that the data presented are sufficient to indicate that, when pavement concrete is to be made of a variety of combinations of aggregates and a reasonably uniform minimum flexural strength is desired, investigations of the materials should be made along the lines indicated in this report in order to predetermine the flexural strengths to be expected. Compressive strength tests on concrete made with the materials to be used are inadequate as the ratio of flexural to compressive strength varies over wide limits for different combinations of materials.

DISCUSSION

Mr. C. E. Proudley.[1]—The investigation reported by Mr. Kellermann proved to be of great value to the Division of Materials and Tests in North Carolina and formed the basis for most of the concrete pavement mixtures used about three seasons ago—during 1937. More recent experience with this method of design as practiced in North Carolina may be of interest.

It was soon discovered that variations in laboratory conditions from day to day would require a standard for strength comparisons. We selected a fine and a coarse aggregate source which gave the most uniform beam test results and, after several preliminary tests, determined the average cement content which would give, practically, the specified design strength of 550 psi. modulus of rupture at 14 days using the lowest testing acceptable portland cement available. Thereafter this mix was used and parallel specimens of it made with each series of tests just as Ottawa sand specimens are used in the determination of sand mortar strength ratios. This has made it possible to duplicate our cement factor determinations with reasonable accuracy as long as the quality of the aggregates and their gradation remained the same.

The use of the lowest testing acceptable cement in the determination of the cement factor was mentioned as a requirement of the specifications. This, of course, should result in ample strength to meet specifications regardless of the source of cement used on a job. Each year we obtain a barrel of cement from each of the mills serving North Carolina and conduct tests to determine the cement giving the lowest strength. Needless to say, these test results are kept confidential. However, the accompanying Table I may be of interest in showing the range of strength obtained. A constant water-cement ratio was used and the consistency, as slump, measured for each batch. The same proportions of fine and coarse aggregates, used as our laboratory standard, were used

TABLE I.—SOME CHARACTERISTICS OF CONCRETE MADE WITH VARIOUS BRANDS OF CEMENT AVAILABLE IN NORTH CAROLINA.

Proportions and materials, except source of cement, were identical for all beams tested. Each value is average of tests on three beams. Cement submitted by mill representatives.

Source	Modulus of Rupture at 14 days, psi.			Consistency, Slump, in.
	Sampled March, 1940	Sampled May, 1940	Average	
A	615	659	622	2.2
B	575	651	613	2.1
C	602	616	609	1.9
D	595	604	600	2.4
E	596	591	594	2.8
F	553	632	592	2.4
G	586	590	588	2.7
H	579	598	588	2.2
I	567	603	585	2.2
J	557	606	582	2.7
K	566	575	570	2.5
L	552	585	568	1.8
M	565	569	567	2.7
N	536	594	565	2.1
O	562	569	564	1.9
P	546	575	560	2.4
Q	554	562	558	2.2
R	564	539	552	2.5
S	511	591	551	2.1
T	522	568	545	2.0
U	518	572	545	2.7
V	518	565	542	2.3
W	509	565	537	2.3

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[1] Engineer of Materials and Tests, North Carolina State Highway and Public Works Commission, Raleigh, N. C.

throughout. Table I shows the change in consistency with brand of cement.

The North Carolina specifications permit the adjustment of cement content to give 550 psi. modulus of rupture at 14 days under laboratory conditions. In other words, if a high testing cement is to be used on the job, a check series of beam-strength tests should be made using the combination of cement, fine and coarse aggregate proposed for use and the cement factor reduced accordingly to effect some saving in cost of cement. Actually this is seldom accomplished, as very few jobs are of sufficient length and the laboratory is not informed sufficiently in advance as to the materials to be used to allow time (approximately 3 weeks) for the tests to be made and be of value.

Beam specimens are made in the field as the work progresses. Two beams are made each day and cured, in so far as possible, the same as the pavement slab. Beams representing five days work are tested at 14 days as indicative of the quality of the concrete actually obtained. Additional beams are usually made and tested at various ages less than 14 days to determine the age at which 550 psi. modulus of rupture is reached, at which age the pavement may be opened to traffic. Field specimens are given no consideration, however, in the determination of cement content requirements.

North Carolina specifications for coarse aggregate for concrete for pavement require gradation from 2 in. to No. 4. For several years we used 6 by 6 by 30-in. molds for laboratory tests, as well as field specimens, although the A.S.T.M. standard method[2] formerly required 6 by 8 by 30-in. molds. Aggregates of uniform particle quality gave satisfactory results, but when materials containing a variety of quality of particles were under examination weird results were obtained on too frequent occasions and the tests had to be repeated. We now use the 6 by 8 by 30-in. molds with far greater uniformity. Also, we had been determining our cement factor – beam strength curve from three points: 1.35, 1.45 and 1.55 barrels of cement per cubic yard of concrete; specimens made on three consecutive days. Now we use four points: 1.30, 1.40, 1.50 and 1.60 in order to establish the shape and position of the curve more definitely.

Mr. Kellermann's report covered only 25 combinations of fine and coarse aggregate. At present we have a published list of 66 and there are about 15 more which have not been published due to the restricted conditions under which they would be used. Several recently developed commercial sand, gravel, and stone deposits have caused us to make and recheck about ten additional combinations in recent weeks. All this, in addition to the necessity for rechecking determinations whenever the method of production at a plant or the quality of the product changes, keeps the concrete laboratory, which we feel is very well equipped, continuously busy.

The lowest cement factor we permit at present is 1.35. Our specifications limit the water content to 6.75 gal. per sack of cement and with a consistency of approximately 2-in. slump the 1.35 minimum cement factor insures fulfillment of specifications. We feel that climatic conditions in North Carolina warrant the values required by our specifications.

It will be observed that very low percentages of sand in the mix were used by Mr. Kellermann. Although it was possible to obtain a satisfactory pavement surface with these relatively undersanded mixtures, we came to feel that too much work was required and the bottom of the slab was probably honeycombed. Cores drilled from the pavement indicated that this was true in many instances. By gradually increasing the percentage of sand we found that no additional water was required to keep the mix workable up to a certain point. We have, therefore, increased our sand content from 2 to 5 per cent above those shown by Mr. Kellermann.

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[2] Standard Method of Test for Flexural Strength of Concrete (Laboratory Method Using Simple Beam with Third Point Loading) (C 78 – 39), 1939 Book of A.S.T.M. Standards, Part II, p. 339.

In our laboratory concrete design work we do not refer to the mortar voids ratio or b/b_o as such. The relationship of the volume of mortar to the volume of concrete or yield is used and found to be very handy when adjusting a mix for cement content. With any given aggregates this mortar-yield ratio and the percentage of water by volume (W_c) are kept constant for variations in cement content. All adjustments are made in the mortar, cement being substituted for sand with an increase in cement factor. The mortar-yield ratio and percentage of water varies with different aggregates depending on the gradation and particle shape of the aggregates. The usual range for the mortar-yield ratio is from 0.50 for gravel coarse aggregate to 0.57 for hard, angular crushed stone. Over the narrow range of cement factors in which we are interested, this method has been found to work satisfactorily. This is largely due to the fact stated in Mr. Kellermann's paper, as noted in publications by Mr. Lyse, that for the same amount of aggregate and a given consistency the water content is a constant regardless of the quantity of cement present. Usually it is only necessary to substitute sand for cement to decrease the cement factor and keep the same consistency.

Job conditions are frequently so different from what was anticipated by the laboratory work that we have a concrete technician who is present when the job is getting started and who has authority to make adjustments in the proportions when necessary. The water-cement ratio is never increased unless laboratory data are available to show that it can be done safely, and even then the 6.75 gal. per sack of cement will not be exceeded. The cement factor is kept constant by adjustments in quantity of aggregate if the water content is varied.

In spite of the numerous difficulties mentioned here, and others not mentioned, I believe that this method is justifiable where a wide variety of types and quality of aggregates must be considered. We do not feel that we have perfected the systematic application of the specification to its greatest effectiveness, but, undoubtedly, the concrete pavements built since the inauguration of these specifications have been steadily improving in so far as quality of concrete is concerned. In this improvement, the producers of materials have been an important factor since, for competitive reasons, each endeavors to produce materials which will give him the lowest cement factor possible as shown on the published tabulation of mixtures for concrete pavement.

THE CHAIRMAN (*Mr. Stanton Walker*[3]).—I was very much interested in the chart which Mr. Jackson used[4] in presenting Mr. Kellermann's paper, which showed a relationship between maximum permissible fineness modulus and the voids in the coarse aggregate. The data are most illuminating and may help to clear up a great many misunderstandings which have been held with respect to the usability of the fineness modulus. I have made detailed use of the fineness modulus since its development some 25 yr. ago and I have always found it a most useful tool. When it has been damned, I have felt that, in most cases, it is not the fineness modulus which is at fault.

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[3] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

[4] Reproduced in the accompanying Fig. 1 of Mr. Kellermann's closure.

I suggest that the relationship shown by Mr. Jackson between voids and maximum permissible fineness modulus is a "secondary" one. The tests on which it is based were made with coarse aggregates of the same grading but of different particle shapes. I believe, therefore, that the voids are a reflection of particle shape and that it is particle shape, rather than voids, which led to the different maximum permissible fineness moduli.

Abrams, in one of his earlier, probably his earliest, published discussions of the fineness modulus recognized that particle shape affected the maximum permissible value. Reference to his famous *Bulletin 1*[5] will show that he recommended differences in maximum permissible values as great as 0.40, depending upon particle shape. Mr. Jackson's data suggest refinements which may be made in the original correction factor.

MR. C. H. SCHOLER.[6]—Needless to say I am delighted at the renewed interest in properly designing concrete mixtures. For many years the Kansas State Highway Commission has designed all concrete mixtures to best fit the use to which the concrete will be put and the materials, cement, sand and coarse aggregate, from which it is proposed to manufacture the concrete. The specifications under which this is done are extremely simple. Depending upon the kind of concrete desired, a minimum cement content and maximum water content per bag of cement are established. In addition, limiting values of a slump are specified.

The minimum cement content specified is low enough so that only those aggregate combinations that approach the ideal can be used with this minimum value. This at once makes it desirable for a contractor to secure not the cheapest material as priced by the ton but the better material which gives the denser, more economical and desirable concrete.

It is very difficult to cover a state of 81,000 square miles with a single aggregate specification that will assure getting the best material in each section of the state. The result is that under the usual standard specifications, with, of necessity, wide tolerances, the competent, well equipped, conscientious producer of aggregate is always at the mercy of the one who just barely scrapes by the limit of the specifications. When a specification for concrete, like the Kansas specification, is in use the contractor profits by buying the better material, the state profits by securing denser, more uniform concrete, and the capable aggregate producer profits by being freed from irresponsible, incompetent competition. Our contractors write more vigorous grading and uniformity specifications than the state could possibly use in the general specifications for the state as a whole.

I believe in the American system of free competition. A proper specification for the design of concrete mixtures will put real competition back into the field of concrete materials for both aggregates and cements.

Properly designed concrete mixtures in which the contractor can share in the profits of a properly designed mixture tends to develop better concrete manipulation and finishing procedure. When a contractor finds that, as he wishes to put in more water to secure concrete easier to handle, he must also, at his own expense, put in added cement, it has a remarkable effect upon his ability to handle drier, more desirable concrete mixtures. He uses equipment and procedures adapted to the type of concrete being produced at the least cost.

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[5] D. A. Abrams, "The Design of Concrete Mixtures," *Bulletin No. 1*, Structural Materials Research Laboratory, Lewis Institute (1919).

[6] Professor of Applied Mechanics, Kansas State College of Agriculture and Applied Science, Manhattan, Kans.

This type of specification is particularly desirable where the quality and availability of materials varies widely in different portions of the state. Because of the wide range of quality and the scarcity of desirable coarse aggregate in certain sections of the state, Kansas regularly places concrete varying in sand content between 28 and 82 per cent.

MR. F. H. JACKSON.[7]—I was interested in what Mr. Scholer said about Kansas. I am inclined to think that most of the troubles there are due to the grading rather than the quality of the materials. Materials in North Carolina and many other states differ greatly in so far as the inherent characteristics of the particles themselves are concerned. In making concrete in these cases it is necessary to investigate the strength of different combinations of materials by determining experimentally the water-cement ratio – strength relation. Kansas has a flat minimum cement factor, a flat maximum water-cement ratio, and any combination within those limits is satisfactory.

I should like to call attention also to one other state where a procedure is used which puts the contractor on his mettle in so far as securing and furnishing the very best materials is concerned. I refer to the state of Texas. Texas goes Kansas one better, because it not only makes it to the contractor's advantage to secure the best materials, but it also determines the mix to be used *after* the contract has been awarded. Prospective bidders must bid on their knowledge of the available materials, based either on prior use or on their own tests. After the contract has been awarded, the state comes in and, using samples of the materials furnished by the contractor, sets up the mix which is to govern. This is done by making trial tests in the field to determine the proper water-cement ratio which must be used for the particular combination of materials selected by the contractor.

MR. H. S. MATTIMORE.[8]—When it was mentioned that it was possible to have many different combinations, the thought might occur that it would be very difficult to proceed with design concrete under such conditions. What I wish especially to bring out is that a number of different combinations are possible under the design concrete specifications now in force in the Pennsylvania Department of Highways work. The conditions encountered proved to be readily solvable by men trained along this line.

I might say that on the Pennsylvania Turnpike the 160 miles of 48-ft. pavement was all design concrete and upon inquiry I was informed that it proved to be a practical procedure and was favored by contractors doing the work.

In carrying out concrete design where a number of different materials are used we found that one subject which has to be given considerable attention by the producers is to keep the aggregates uniform so far as grading is concerned.

Another factor that has to be given consideration is a uniformity in moisture content, especially in the fine aggregate.

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[7] Senior Engineer of Tests, Public Roads Administration, Federal Works Agency, Washington, D. C.

[8] Engineer of Materials, Pennsylvania State Highway Dept., Harrisburg, Pa.

We found that this can be controlled largely by having this fine aggregate stocked from 18 to 24 hr.

An interesting phase of material control in design concrete is that under specifications of this type the contractor recognizes the advantage of uniformity in grading. We found that he is impressing this fact on the material producer and the result has been that complaints along this line formerly made by the engineers to the material producers are taken up by the contractor from the standpoint of producing a uniform concrete.

One procedure sometimes followed in the production of sand is the utilization of crushed particles of gravel of the fine aggregate size. When we are operating with design concrete this has proved to be quite troublesome from several standpoints. One is that the particle shape of the crushed aggregate varies from the natural sand and affects the necessary water content. This is especially aggravated in that the amount of coarse particles varies in daily production. We have found that the real solution is to prevent the addition of such crushed particles through the sand as a routine procedure. If these are to be added it should be specially stocked so that the amount added can be actually under control.

The point that I wish to emphasize especially is that concrete control in highway construction is a practical procedure resulting in uniformity in concrete roads so far as consistency and general condition of the concrete are concerned and in the end should be an economical procedure.

MR. WILLIAM S. ELLIOTT.[9]—The gentleman from North Carolina, in discussing Mr. Kellermann's paper, has, in my opinion, invited Committee C-1 to write another standard specification. Mr. Proudley states that when a specification calls for a constant water-cement ratio in various concrete mixes, it is necessary with some portland cements to put in more cement than with others. If that is true, I believe that we should have a measure of the effect of portland cement on the workability of concrete. This measure is of vital importance to the consumer of portland cement.

MR. W. F. KELLERMANN[10] (*Author's closure by letter*).—The chart referred to by Mr. Walker in his discussion was in the paper as originally written but was later withdrawn because it did not cover a wide range in maximum sizes of coarse aggregates and also because it was based on mixes which, judged by some standards, would be considered as undersanded. It was prepared from data resulting from the analysis of the mix design shown in the report.

In reproducing it here (accompanying Fig. 1), the author does not claim that this chart has a wide practical application. The relationships shown were obtained experimentally, using 13 different coarse aggregates having different shaped particles but all graded alike, combined with 7 different fine aggregates, all graded differently, to give a total of 25 combinations of materials. This, of course, does not begin to cover the field of possible combinations of materials met with in practice. The purpose of the chart is to show how the percentage of sand used in the different mixes was related to the voids in the coarse aggregate. While the relationship shown is between percentage of voids in the coarse aggregate and maximum permissible fineness modulus, the sand percentage is readily calculated by the formula given once the maximum permissible fineness modulus has been determined.

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[9] Engineer of Tests, New York City Department of Public Works, New York, N. Y.

[10] Materials Engineer, Public Roads Administration, Washington, D. C.

The fineness modulus method, as given in modern literature, provides a method of determining the percentage of sand by giving values for the maximum permissible fineness modulus. These values are given for rounded aggregate and an arbitrary correction factor is applied where angular aggregates are used. Thus stress is laid upon the particle shape of the coarse aggregate. In Mr. Walker's discussion he also stresses particle shape and suggests that the void relationship shown by me is perhaps a secondary one. The author feels that, while particle shape may be the "cause," percentage of voids is the "effect" and is therefore the more important consideration because it is something that can be translated into actual values for practical purposes as I have done. Furthermore, it is felt that the fineness modulus method, presented on the basis of a given value for percentage of voids in the coarse aggregate, with correction factors for other void contents, would be a far more useful tool than the method as it stands today. A further refinement could be made by taking cognizance of the voids in the fine aggregate.

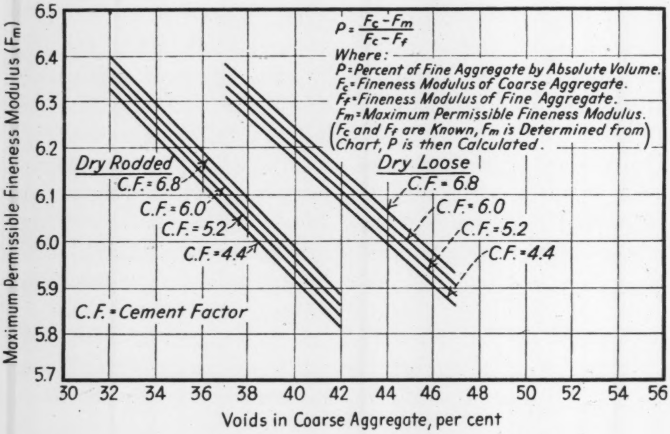

FIG. 1.—Chart for Determining the Ratio of Fine Aggregate to Total Aggregate in a Concrete Mix of 2 to 3-in. Slump. Maximum size of concrete aggregate, 2 in.

In conclusion, it may be stated that had this chart been available at the time the tests were started, much time and effort would have been saved in designing the 125 different mixes.

A METHOD OF MEASURING THERMAL DIFFUSIVITY AND CONDUCTIVITY OF STONE AND CONCRETE

By W. T. Thomson[1]

Synopsis

In dealing with thermal stresses resulting from the transient flow of heat through a body, a term which is of particular importance is the thermal diffusivity. This paper is concerned with one method of determining the thermal diffusivity of materials such as stone and concrete. The theoretical basis for the method is clearly stated and experimental results presented for various stones.

The reason why some aggregates produce durable concrete while others do not has been a subject of wide discussion. It is known that thermal stresses play an important part in the deterioration of concrete. For a given body with specified boundary conditions, the thermal stresses depend on certain physical properties of the material. In a homogeneous body, some of these physical properties such as the thermal conductivity, specific heat, and the density of the material influence the temperature distribution and the thermal stresses during the transient period only in a certain combination known as the thermal diffusivity. The thermal diffusivity is defined as the thermal conductivity divided by the specific heat and density, and is a physical property of the material which determines the time rate of change of temperature of any point within a body.

If in a mixture such as concrete, the thermal diffusivities and conductivities of the aggregate and the cement paste are equal to each other, the body can be thought of as being thermally homogeneous during the transient period as far as temperature distribution is concerned. Since a difference in diffusivities would result in different rates of diffusion of heat through the aggregate and cement, it is believed that such a combination would result in additional thermal stresses over that of homogeneous bodies. Thermal stresses also depend on the modulus of elasticity, thermal coefficient of expansion and Poisson's ratio.

Just what effect such differences in thermal properties produce in the deterioration of concrete can probably be determined only by experimentation and is being investigated by L. V. Nothstine.[2] However, in order to investigate such effects, the thermal constants must be carefully determined for the various aggregates and cement. It is with this in mind that the author wishes to present a practical method of determining directly the thermal diffusivities of such materials, and consequently, an indirect determination of the conductivity.

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[1] Assistant Professor, Department of Applied Mechanics, Kansas State College, Manhattan, Kans.

[2] Graduate Assistant, Department of Applied Mechanics, Kansas State College, Manhattan, Kans. (1939-1940).

Being a ratio of measurable physical properties, the thermal diffusivity can be indirectly calculated or it may be directly determined by experiment. The difficulty of accurately measuring the conductivity of material such as stone favors the direct method of determining the diffusivity. In an article appearing in the *Journal* of the American Concrete Institute, Glover[3] presents one method of determining the diffusivity directly. Diffusivity and conductivity of the concrete used in several dams have also been published.[4] In Glover's method, a cylindrical specimen with length equal to twice the diameter was suddenly cooled in water after the specimen had reached a uniform-higher temperature. From the temperature-time relation of a point at the center of the cylinder, the diffusivity constant was determined by the use of curves based on the mathematical solution of heat transmission. These solutions are dependent on the Biot's modulus which is an indication of the turbulence as well as the nature of the cooling medium, and one is justified in using curves representing a given solution only when the same Biot's modulus can be duplicated. In the following section is given the development of a method whereby the calculation of the thermal diffusivity is independent of the Biot's modulus.

THEORY

In the mathematical theory of heat conduction of a homogeneous body of constant thermal properties, the temperature distribution during the transient period is a function of the following:

1. Initial temperature distribution.
2. Biot's modulus.
3. Fourier's modulus.
4. Some ratio of dimensions depending on the shape of the body.

The Biot's modulus is defined as

$$B = \frac{k'}{l'}\,\frac{l}{k}$$

where k' and l' = equivalent conductivity and equivalent thickness of a film surrounding the body,
k = the conductivity of the body, and
l = some linear dimension of the body, depending on the shape.

The Fourier's modulus is defined as

$$F = \frac{a\theta}{l^2}$$

where a = the thermal diffusivity of the material,
θ = the time, and
l = some linear dimension of the body.

Both the Biot's modulus and the Fourier's modulus are nondimensional and can be used for similarity studies.

The following mathematical basis is given for cylindrical specimens:

Notation:

t = temperature in degrees Fahrenheit,
θ = time in hours,
$a = \frac{\mathrm{k}}{c\rho}$ = thermal diffusivity in square feet per hour,
k = thermal conductivity of the material in Btu. per square feet per hour per degrees Fahrenheit per foot,

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[3] R. E. Glover, "Flow of Heat in Dams," *Journal*, Am. Concrete Inst., Vol. 6, No. 2, November-December, 1934, pp. 113-125.

[4] R. F. Blanks, "Boulder Dam Cement and Concrete Studies," *Engineering News-Record*, Vol. 113, No. 21, November 22, 1934, pp. 648-651.

c = specific heat in Btu. per pound per degree Fahrenheit,
ρ = density in pound per cubic foot,
r = radial distance from center of cylinder in feet,
R = radius of cylinder in feet,
$B = \frac{k'}{l'}\frac{R}{k}$ = Biot's modulus,
$F = \frac{a\theta}{R^2}$ = Fourier's modulus,
T_0 = initial temperature of specimen, and
J_0 = Bessel's function of zero order.

The general equation of heat transmission for an isotropic material of constant conductivity and specific heat, when transformed into cylindrical coordinates where the temperature is a function only of the radius and time, is[5]

$$\frac{\delta t}{\delta\theta} = a\left\{\frac{\delta^2 t}{\delta r^2} + \frac{1}{r}\frac{\delta t}{\delta r}\right\} \ldots\ldots (1)$$

The solution of the above equation is known to be,[6]

$$t = T_0 \sum_{n=1}^{\infty} A_n\, e^{-\lambda_n^2\left(\frac{a\theta}{R^2}\right)} J_0\left(\lambda_n \frac{r}{R}\right) \quad (2)$$

For the special case of a cylinder of uniform initial temperature cooled in a constant temperature medium, λ_n are the roots of the boundary equation[7]

$$\lambda_n\, J_1(\lambda_n) = B\, J_0(\lambda_n) \ldots\ldots (3)$$

and the values of A_n become[8]

$$A_n = \frac{2B}{[B^2 + \lambda_n^2]\, J_0(\lambda_n)} \ldots. (4)$$

Since λ_n is a function only of B, it is apparent that A_n is dependent only on B or the boundary condition.

Given the temperature distribution t/T_0 as a function of time and coordinate, the difficulty of obtaining a numerical value of the diffusivity is evident even if B is known accurately, a case which is seldom true. However, a numerical solution is possible if the series is made highly convergent by a proper choice of the Fourier modulus. For instance, at the center $r = 0$, the series can be written as:

$$t_c = T_0 \sum_{n=1}^{\infty} A_n\, e^{-\lambda_n^2\left(\frac{a\theta}{R^2}\right)} \ldots. (5)$$

In this expression, the ratio of the second term to the first can be made as small as one wishes. Letting p be the ratio of the two terms, the corresponding Fourier modulus becomes

$$F = \left(\frac{a\theta}{R^2}\right) = \frac{1}{\lambda_1 - \lambda_2} \log_e\left(p\,\frac{A_1}{A_2}\right) \quad (6)$$

From this equation it is possible to determine the Fourier's modulus necessary to obtain a degree of convergence p of this series. For any point other than the center, the percentage error p in omitting all terms other than the first is smaller by the factor

$$\frac{J_0\left(\lambda_2 \frac{r}{R}\right)}{J_0\left(\lambda_1 \frac{r}{R}\right)}$$

Referring to Eq. 2, and assuming that the value of F is chosen sufficiently large to reduce the series to a single term with a percentage error less than p, the following relations are found:

For any given time θ_1

$$\left(\frac{t_R}{t_c}\right)_{\theta=\theta_1} = J_0(\lambda_1) \ldots\ldots (7)$$

where t_c and t_R are the temperatures at the center and surface, respectively. Equation 7 enables the determination of

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[5] H. S. Carslaw, "Fourier Series and Integrals, p. 312, Macmillan and Co., New York, N. Y. (1906).
L. M. K. Boelter, "Heat Transfer," p. III-20, University of California Press (1936).

[6] H. S. Carslaw, "Fourier Series and Integrals," p. 314, Macmillan and Co., New York, N. Y. (1906).

[7] *Ibid.*, pp. 327-328.

[8] W. E. Byerly, "Fourier Series and Spherical Harmonics," p. 229, Ginn and Co., New York, N. Y. (1895).

λ_1, which can be found in any table of Bessel's function. Biot's modulus can then be found from Eq. 3 or from Fig. 1.

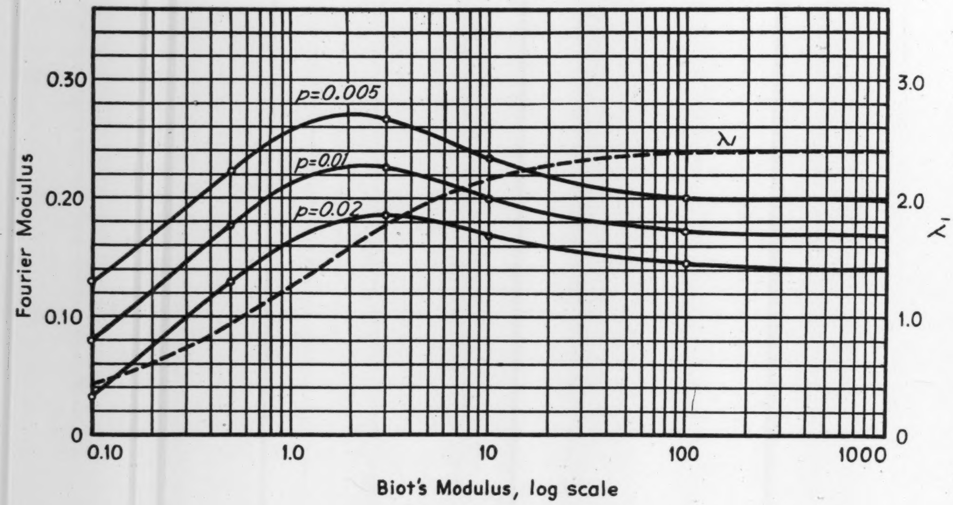

FIG. 1.—Relations for Convergence of Series.

TABLE I.—TABULATED VALUES FOR FIG. 1.

B	λ_1	λ_2	A_1	A_2	$(F)_{\frac{1}{2}\%}$	$(F)_{1\%}$	$(F)_{2\%}$
0.1.......	0.44168	3.858	1.025	−0.0334	0.128	0.0805	0.0335
0.5.......	0.940	3.959	1.13	−0.1574	0.225	0.178	0.132
1.0.......	1.2558	4.0795	1.208	−0.290	0.258	0.213	0.165
3.0.......	1.7885	4.463	1.42	−0.631	0.268	0.227	0.185
10.0.......	2.1795	5.033	1.56	−0.958	0.234	0.200	0.167
100.0.......	2.381	5.465	1.60	−1.064	0.203	0.174	0.145
∞	2.4048	5.520	1.602	−1.066	0.198	0.170	0.142

Taking two different times θ_1 and θ_2 and the corresponding temperatures at the surface, the expression for the diffusivity becomes

$$a = \frac{R^2}{\lambda_1^2 (\theta_2 - \theta_1)} \log_e \left(\frac{t_{\theta_1}}{t_{\theta_2}} \right)_{r=R} \quad . . (8)$$

It is evident here that the calculation of B does not enter into the determination of the diffusivity; however, in order to use the above expression, the value of F must be properly chosen to insure the necessary convergence of the series. For this purpose, the values of A_1 and A_2 were determined from the values of λ_1 and λ_2 representing the solution of Eq. 3.[9] These were tabulated in Table I, and curves showing the relationship of F to B for a given p are given in Fig. 1.

As an illustration, suppose it is desired to reduce the series of Eq. 2 to a single term without introducing an error greater than 1 per cent, thereby enabling the use of Eqs. 7 and 8. The procedure is to first make a rough calculation, after which an accurate calculation can be made.

From Fig. 1, regardless of the value of B, if F is chosen greater than 0.23, the percentage error in disregarding the higher terms of the series will be less than 1 per cent. Assuming an approximate lower limit of the value a, the time θ for any cylinder can be found. For any time greater than this, Eqs. 7 and 8 can be used, and the percentage error in neglecting the other terms in the series will be less than 1 per cent. A recalculation with the value of a found can then be made.

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[9] Gerald Pickett, "The Effect of Biot's Modulus on Transient Thermal Stresses in Concrete Cylinders," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 913 (1939).

Another method which accomplishes the same result is to find the time at which $\left(\frac{t_R}{t_c}\right)_{\theta_1}$ becomes constant. For any time greater than this, Eqs. 7 and 8 can be used with negligible error.

From Eq. 6, and Fig. 1 the following important conclusions can be made:

1. The series converges at a more rapid rate for small values of B. (That is, it is better to cool the specimen in air than in water.)
2. The series converges at a more rapid rate for small values of R, therefore, a smaller cylinder is preferable to a larger one.
3. The series converges at a more rapid rate for materials of large diffusivity.

These conclusions have been helpful in guiding the experimental work, some of the results of which are given in the following sections.

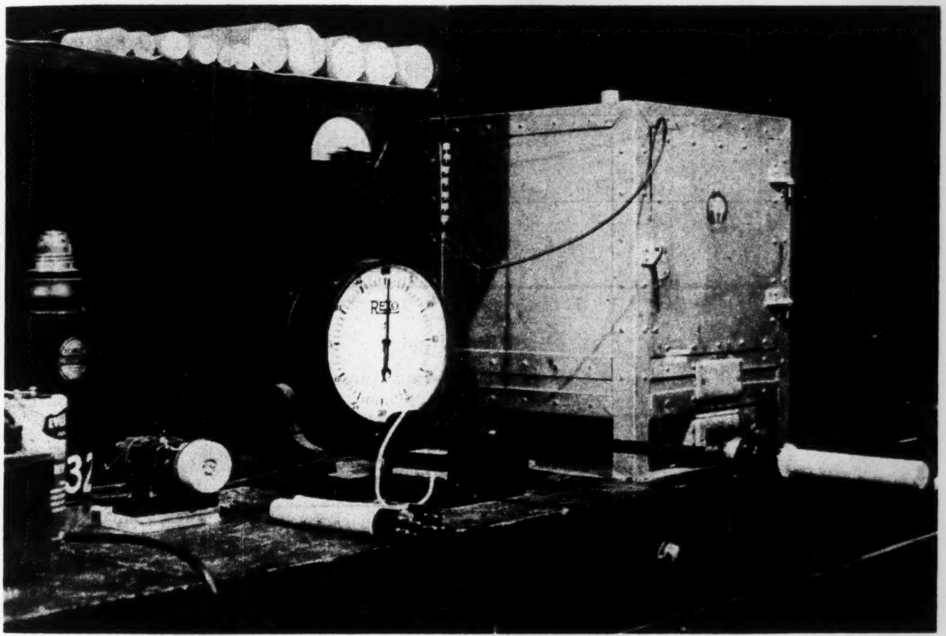

FIG. 2.—Apparatus for Diffusivity Determination.

APPARATUS AND TEST PROCEDURE

The cylindrical specimens used in these tests were obtained by core drilling various stones, the dimensions of the cylinders being approximately 1 by 8 in. Copper and constantin thermocouple junctions of No. 28 wire were used; one at the center and the other just under the outer surface. In placing the thermocouple, a small hole was drilled in the specimen and filled with neat cement after the junction was placed in the proper position. The specimens were mounted in old radio tube bases, and the thermocouple circuits made through the plug-in socket and slip rings rotating in mercury pools. These mercury contacts were found to be free from parasitical voltages. The whole assembly was rotated by a small motor to produce a symmetrical cooling of the specimen with a unidirectional stream of air from an electric fan. The arrangement of the apparatus is indicated in Fig. 2.

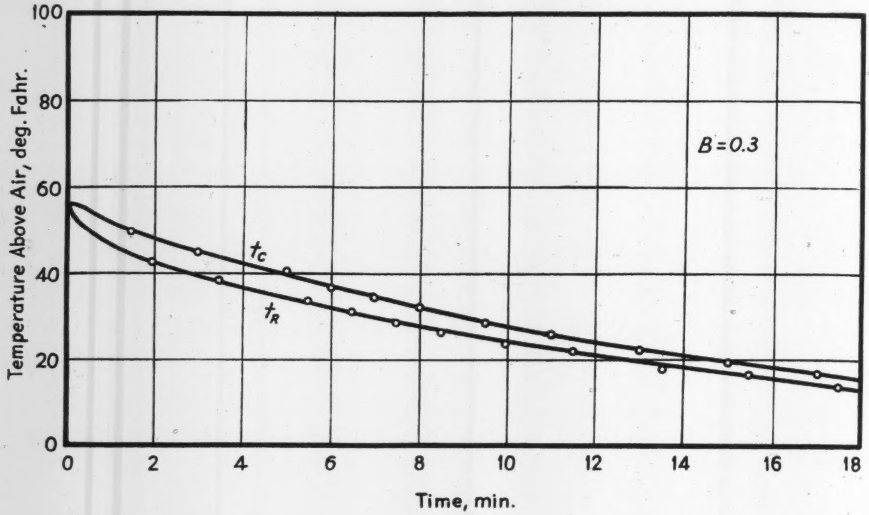

FIG. 3.—Cooling Curve of Fossiliferous Limestone at $B = 0.3$.

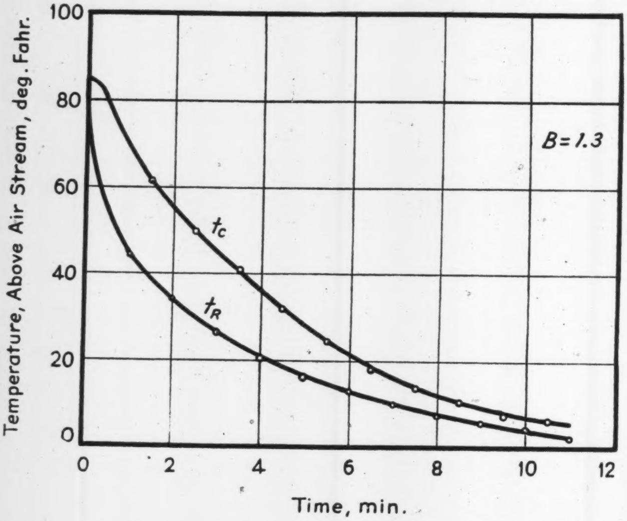

FIG. 4.—Cooling Curve of Fossiliferous Limestone at $B = 1.3$.

In performing the test, the specimen was heated in an electric oven for a sufficient length of time to insure uniform temperature throughout. A second socket connected to the specimen while in the oven indicated the temperature of the specimen during the heating period. An initial temperature of approximately 180 F. was used for most of the specimen. As the specimen was taken out of the oven and plugged into the rotating socket, the clock was started and the temperature of the center and outer surfaces was recorded every half minute during cooling. Various rates of cooling were obtained by adjusting the speed and the distance of the fan from the specimen.

EXPERIMENTAL RESULTS

Several tests were performed on each specimen at different Biot's modulus. Figures 3 and 4 represent typical cooling curves for a given specimen at different Biot's modulus. In order to illustrate the method of determining the diffusivity, the following calculations are presented:

TABLE II.—THERMAL CONSTANTS OF STONES.

Material	Origin	Specific Gravity	a	c	k
Sandstone	Lincoln County, Kans.	2.64	0.0270	0.173	0.770
Fossiliferous limestone	Riley County, Kans.	2.74	0.0137	0.169	0.396
Serpentine	Bala, Kans.	2.68	0.0197	0.203	0.668
Limestone	Ohio	2.60	0.0317	0.180	0.924
Neat cement cylinders	Victor brand (Portland cement)	2.04	0.0126	0.222	0.357

Specimen—Fossiliferous limestone—$R = \frac{1}{2}$ in.

θ, min.	t_c	t_R	$\left(\frac{t_R}{t_c}\right)$
5	39.4	34.2	0.867
6	36.8	32.0	0.870
7	34.3	29.5	0.860
8	31.9	27.3	0.856
9	29.4	25.3	0.867
10	27.3	24.0	0.879
		Avg. =	0.866

$J_0(\lambda_1) = 0.866$ from Eq. 7.

$\lambda_1 = 0.744$ from tables of Bessel's functions.

$B = 0.30$ from Fig. 1.

Using the temperatures at 5 and 10 min., and substituting into Eq. 8

$$a = \frac{\left(\frac{1}{24}\right)^2}{0.744^2\left(\frac{5}{60}\right)} \log_e\left(\frac{34.2}{24.0}\right)$$

$$= 0.0133 \text{ sq. ft. per hr.}$$

From calorimeter test specific heat = $c = 0.1691$ Btu. per lb. per deg. Fahr.

Density = 2.738

$\therefore k = a\rho c = 0.0133 \times (2.74 \times 62.4) \times 0.1691 = 0.384$ Btu. per sq. ft. per hr. deg. Fahr. per ft.

The same specimen when tested at $B = 1.3$ (see Fig. 4) gave a value of diffusivity equal to 0.0138. For any given type of rock, the diffusivities found by this test were within 10 per cent of the average.

The average values of the thermal constants calculated by this method are presented in Table II.

CONCLUSIONS

In developing the method described in this paper, a number of difficulties were encountered. Since the accuracy of results obtained depends mainly on these factors, they will be briefly described.

1. The thermocouples must be of fine wire to prevent additional conduction through the wire.

2. The thermocouple on the outside

of the specimen must be placed slightly under the surface and must not be exposed.

3. The temperature of the cooling air must be kept constant, a variation of this temperature causing a large variation of the diffusivities calculated. The difficulty can be overcome by using a large room.

4. Cooling in water was not used due to the penetration of the water into the specimen during the test.

5. Specimens obtained from different parts of the same rock may give different results due to difference in grain of the rock.

With these precautions in mind, and by careful experimentation, the thermal constants of many materials can be determined with acceptable accuracy. Compared to the method of calculating the conductivity directly, this method of determining the thermal constants is of great advantage.

In closing, the author wishes to acknowledge thanks to L. V. Nothstine for his assistance in obtaining the experimental results presented in this paper.

DISCUSSION

MR. L. M. K. BOELTER[1] (*presented in written form*).—The determination of thermal constants from the measurements of thermal variables during the transient period deserves much greater attention than has been given to it heretofore. Obviously the system used must be idealized to conform with one of the mathematical solutions available.

Mr. Thomson presents a method of determining the thermal diffusivity (and indirectly the thermal conductivity) which does not require the evaluation of the thermal gradients in the specimen. The transient method has also been applied successfully for the determination of the unit thermal conductance between a body and its surroundings by A. L. London, Instructor of Mechanical Engineering, Stanford University, Palo Alto, Calif.

MR. GERALD PICKETT.[2]—Mr. Thomson is to be commended for recognizing first the existence of a surface-film factor and then developing a technique whereby the value of the surface factor has negligible effect upon the determination of thermal diffusivity. In the majority of scientific papers devoted to the effects of transient heat or moisture flow in solids, the problem is idealized and thereby mathematically simplified by the tacit assumption that the Biot modulus is infinity. This is rarely justifiable.

In the interest of scientific accuracy I should like to point out that two materials must have equal values of conductivity as well as equal values of thermal diffusivity in order that a combination of the two materials may give a thermally homogeneous body. To illustrate this necessity, let two materials A and B have the same thermal diffusivity but let the conductivity of A be twice as great as the conductivity of B. Let three 4-in. slabs be made, the first of material A, the second of material B, and the third have the central 2 in. of material A with 1 in. of material B on either side. Let the three slabs be heated to the same equilibrium temperature and then cooled. Given the same surface factors in all cases, the first two slabs will cool alike but the third will cool more slowly because the material with the high heat per unit volume is surrounded by the material of low conductivity.

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THE THEORY OF CONCRETE CREEP

BY WILLIAM R. LORMAN[1]

SYNOPSIS

This paper is written with the view of establishing a simple and practicable method for determining the creep of plain concrete subjected to sustained load.

The phenomenon of creep is defined and briefly described. The explanation of the manner in which creep and shrinkage occur is based on the gel theory as commonly accepted today. For constant sustained load the creep-time relation is expressed by a hyperbolic type of equation which is demonstrated as being quite feasible in predicting the creep of concrete. The creep-time equations for variable sustained load are derived from the above hyperbolic equation which is considered the basic law of creep. Also presented are two methods of determining the creep constants in the equations, the method used being dependent upon whether the sustained load is constant or variable.

Correlation of experimental data found in the literature on concrete creep has made possible the establishment of the fundamental principles concerning the manner in which the creep constants are modified by various physical factors. Among these factors governing the magnitude of creep *mix proportions* and *aggregate gradation* are believed most important. Indications are that within the range of plastic mixes creep is governed by the water-cement ratio and is practically independent of the cement content of the mix. Study of available creep data also points to the conclusion that the creep coefficient, which is numerically equal to the ultimate creep strain per unit of stress, increases with the fineness modulus of the aggregate.

Concrete creep is the nonelastic deformation, which continues to increase with time, of concrete subjected to sustained load. Throughout this paper concrete creep is considered as an inelastic deformation due entirely to the action of sustained load and is not to be construed as including any deformation due to other causes such as growth, shrinkage, swelling, etc. All creep data used in establishing the theory herein are in conformity with the above interpretation. Creep is desirable in concrete structures since the favorable distribution of stresses produced by volume changes depends upon the ability of the concrete to adjust itself to stress conditions. The effect of creep is in general to relieve the stresses and thus aid in reducing the tendency toward cracking.

Probably the first reference to creep of concrete is that regarding the experiments of Woolson **(21)**[2] in 1905. As far back as 1925, when the Stevenson Creek Arch Dam investigation was projected, sufficient facts concerning concrete creep had previously been established to warrant inclusion of this phenomenon as an important factor. Accordingly, Davis (2) was commissioned to perform laboratory tests dealing with creep of concrete. The results were valuable in that they aided in reaching a satisfactory explanation of the volume changes occurring in the concrete of the dam. In 1927 Faber (10) published his paper showing creep as an important factor in reinforced concrete design. Soon after, Glanville (12) began a series of laboratory experiments and subsequently introduced what perhaps were the first theoretical calculations involving the properties of creep. Both American and British investigators (Ross (15), Shank (17), and Thomas (19)) have since that time published the findings of their observations together with suggested mathematical expressions for predicting the amount of creep taking place in a concrete subjected to certain conditions.

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[1] Assistant Engineer, Bureau of Reclamation, United States Department of the Interior, Denver, Colo.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1102.

Despite the seemingly large amount of work accomplished by various investigators, the experimental data published during the past few years are limited in scope. This is due to the large number of physical variables involved and to the lack of correlated experimental results of various investigations. Thus much difficulty is encountered in estimating the creep anticipated to occur in a given concrete. It is the intention of the author to develop methods whereby one may obtain a more exact determination of the magnitude of creep. With slight changes, Ross' (15) type of creep-time relation has been the basis for the concrete creep theory as presented in this paper.

It is hoped that this contribution will point to the great need for correlated experimental data dealing with creep of concrete. At present, any efforts to correlate the findings of various experimenters invariably are met with considerable difficulty.

THE GEL THEORY

It has been suggested that creep of concrete may involve all three of the following types of yielding: (*a*) *crystalline flow* (in a crystalline mass, slippage along planes within the crystals); (*b*) *seepage* (due to applied pressure, flow of adsorbed water from the cement gel); and (*c*) *viscous flow* (movement of particles, as in the flow of asphalt). A portion of the creep possibly may be due to crystalline or viscous flow; nevertheless, it is believed that the major portion is caused by seepage, which would appear to be the most acceptable explanation of creep. The hydration of portland cement results in the formation of an amorphous or gelatinous mass, ordinarily termed "gel," which serves to connect the aggregate particles. Water may exist in the concrete mass in three principal forms (11): (*a*) *chemically-combined water* (in chemical combination with the cement), (*b*) *adsorbed water* (adsorbed by the cement gel), and (*c*) *free water* (water within the microscopic pores or spaces between the gel particles). According to Lynam (14), chemically-combined and free water play no direct part in volume changes. Thus, except for the effect of hydration, gain or loss of adsorbed water from the gel appears to be the basis of volume changes resulting from ambient moisture variations or from sustained pressure. The gel may be considered as having microscopic pores; with the removal of water the pore spaces collapse and the gel shrinks, while upon the addition of moisture the pore spaces adsorb water and the gel expands. This process is dependent upon frictional resistance to flow of water along the capillary channels which permeate the mass of concrete. Other things being equal, the total frictional resistance is governed by the moisture gradient. The steeper the moisture gradient, the easier the flow of water through the capillary channels. Volume change of the gel may, on the other hand, be dependent upon seepage caused by applied external pressure. Subjecting the concrete to an external load, the adsorbed water is expelled from the gel. The rate of expulsion of moisture in this instance is a function of the applied load and of the friction in the capillary channels. The greater the applied load, the steeper the pressure gradient with consequent increase in rate of moisture expulsion. By the foregoing hypothesis shrinkage or swelling due to loss or gain of moisture and creep due to seepage are interrelated phenomena. Despite this relation the two are considered separately.

CREEP OF CONCRETE SUBJECTED TO CONSTANT SUSTAINED LOAD

Hooke's Law for Creep Strain:

Results of the more recent laboratory tests point to the conclusion that, within the range of design stresses, creep strain or unit creep is proportional to the stress. The creep-stress relation for constant sustained load may be written in the form

$$\epsilon_c = f(t)\cdot\sigma \qquad (1)$$

where ϵ_c is the creep strain in millionths of an inch per inch, σ is the stress in pounds per square inch, and the coefficient $f(t)$ is a function of time because creep strain varies while the stress remains constant. From test results published by Glanville (13) and by Ross (16) it appears that Hooke's law applies to creep strain accurately for stresses from 0 to 650 psi. and approximately for stresses from 650 to 1500 psi.

Creep-Time Equation:

Creep-time curves which have been carried sufficiently far clearly indicate that over a long period of time creep approaches a limiting value. The limiting value toward which unit creep tends under a given set of conditions is termed ultimate creep strain. During the past decade various types of creep-time equations have been proposed by American and British authors. It is apparent that only those equations which make creep tend toward a finite limit are of practical value. Probably the most outstanding contributions in this respect are a hyperbolic form proposed by Ross (15) and an exponential form developed by Thomas (19), each asymptotically approaching a constant value. In this paper the hyperbolic expression will be used exclusively because of its comparative simplicity.

Assuming $f(t)$ in Eq. 1 in the hyperbolic form, one has

$$\epsilon_c = \frac{mt}{n+t}\cdot\sigma \qquad (2)$$

where t is the time in days reckoned from the instant when the load is applied, and m and n are constants whose significance is explained below. When t becomes very large, the quotient $\frac{t}{n+t}$ approaches unity. It follows that the limiting value of creep strain is

$$\epsilon_c = m\sigma$$

where m is the *creep coefficient* in millionths of an inch per inch per pound per square inch and is numerically equal to the ultimate creep strain per unit of stress. Setting t equal to n, one has

$$\epsilon_c = \frac{m}{2}\cdot\sigma$$

whence it follows that 50 per cent of the ultimate creep strain is obtained at the time $t = n$. Since the value of n governs the rate at which the limiting value of creep is approached, n is termed *creep-time constant* and is measured in the same units as time (in this case, days). The creep coefficient m and the creep-time constant n collectively will be referred to as the *creep constants.*

Determination of Creep Constants:

When the relation between creep and time for a specific case is given in the form of a curve or a numerical table, the creep constants may be determined by the following graphical solution. If the data are such that they can be fitted by the hyperbolic creep-time equation the resulting graph is a straight line. The slope of the straight line is equal to m and the vertical intercept is numerically equal to n.

The following example will serve as an illustration of the method. Required to find m and n for the creep-time curve in Fig. 1 (insert). Knowing that $\sigma = 800$ psi. and taking related values of t and ϵ_c from the curve, calculate v. Plot t against v as shown in Fig. 1. Pass a straight line through the points, attaching more significance to points farther to the right so as to obtain more accurately the ultimate value of creep strain. Measure the slope and the vertical intercept, obtaining the creep coefficient $m = 0.36$ and the creep-time constant $n = 15$. The fact that the points in the graphical solution in Fig. 1 practically lie on a straight line indicates

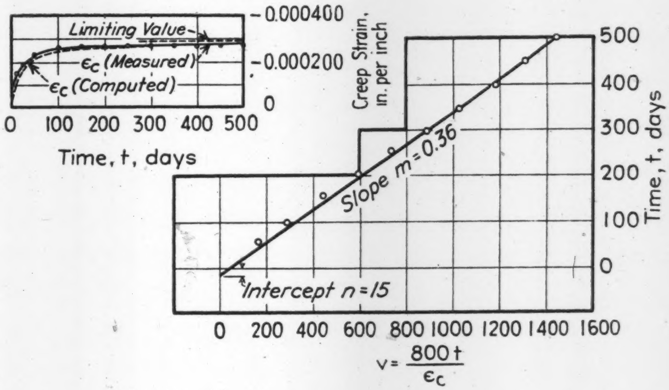

FIG. 1.—Graphical Solution for Creep Constants.

Diameter of specimens—6 in.
Aggregate—gravel (source unknown).
Maximum size of aggregate—1½ in.
Combined fineness modulus—5.69.
Cement—normal portland.
Aggregate-cement ratio by weight—6.95.
Water-cement ratio by weight—0.61.
Slump—unknown.
Curing—28 days, water, 70 F.
Age at loading—28 days.
Sustained stress: 800 psi. compression.
Storage after loading—100 per cent relative humidity, 70 F.
Source—R. E. Davis, H. E. Davis, and J. S. Hamilton, "Plastic Flow of Concrete Under Sustained Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 368, (Fig. 7, Series 13) (1934).

Clearing fractions in Eq. 2 one has

$$(n + t)\,\epsilon_c = m\sigma t \ldots\ldots\ldots\ldots(3)$$

Introducing the quantity v defined by the relation

$$v = \frac{\sigma t}{\epsilon_c}$$

and solving Eq. 3 for t, one has

$$t = mv - n$$

Note that t is a linear function of v, since m and n are constants. From the creep data, values of v may be calculated and t may be plotted against v.

that the creep data given by the solid curve in the insert can be closely represented by the hyperbolic creep-time equation. For visual comparison, Fig. 1 (insert) shows the calculated creep strain plotted alongside the measured creep strain. Experience of the author has proven that creep data obtained by Davis, Glanville, Ross, Thomas, and other investigators can be fitted by the hyperbolic creep-time equation with an accuracy adequate for all practical purposes.

CREEP OF CONCRETE SUBJECTED TO VARIABLE SUSTAINED LOAD

Derivation of Equations:

In the preceding section only that case was considered where the entire stress becomes effective at once. Of importance also is the case where the stress is applied gradually (in increments or continuously). A solution for the case of gradual loading ordinarily can be obtained by a process of summation since the effects of component stresses are superposable as long as the total stress does not exceed the limits of Hooke's law for creep strain. It will be assumed throughout this section that the total stress does not exceed 650 psi.

Loads which are applied during an interval of several seconds or less will be regarded as instantly applied loads. The effective time of application of the load may be taken as the instant when two-thirds of the load has been realized. However, it is seldom necessary to reckon time closer than to the nearest day. If a constant sustained load becomes instantly effective at the time $t = 0$, the creep strain produced is

$$\epsilon_c = \frac{mt}{n+t}\cdot\sigma$$

where time is reckoned from the instant when the load is applied. If the load is applied at the time $t = \tau$, that is, if time is reckoned from an instant prior to the application of the load, the relation becomes

$$\epsilon_c = \frac{m(t-\tau)}{n+(t-\tau)}\cdot\sigma, \qquad t \geqq \tau \ldots\ldots (4)$$

where τ is the time of application of the load.

Suppose the stress is applied in two separate increments. Stress increment $\Delta\sigma_1$ becomes instantly effective at the time $t = \tau_1$; stress increment $\Delta\sigma_2$, at the time $t = \tau_2$. The entire stress is

$$\sigma_0 = \Delta\sigma_1 + \Delta\sigma_2$$

The creep constants generally vary with the age of the concrete at the time of load application and therefore it becomes necessary to use two sets of constants. Let the creep constants be m_1 and n_1 for creep produced by the first stress increment, m_2 and n_2 for creep produced by the second increment. By virtue of Eq. 4 the creep strain at any time after the application of the second stress increment is

$$\epsilon_c = \frac{m_1(t-\tau_1)}{n_1+(t-\tau_1)}\,\Delta\sigma_1 + \frac{m_2(t-\tau_2)}{n_2+(t-\tau_2)}\,\Delta\sigma_2, \qquad t \geqq \tau_2 > \tau_1 \ldots (5)$$

If a large number P of stress increments are applied at regular time intervals and if the rate of loading is known, the creep-time relation is expressed in the form

$$\epsilon_c = \sum_{x=1}^{x=P} m_x \frac{t-\tau_x}{n_x+(t-\tau_x)}\cdot\frac{\Delta\sigma_x}{\Delta\tau_x}\,\Delta\tau_x, \qquad t \geqq \tau_P \ldots (6)$$

A study of creep data indicates that if the concrete is more than 90 days old at the time when the first load increment is applied, the creep coefficient is sensibly the same for each term of the summation and may be factored outside the summation sign. Under these conditions n is also the same for each term of the summation. The case where m and n are variable will not be considered.

Suppose the load continuously increases from the time $\tau = 0$, when it is zero, to the time $\tau = T$, when it becomes constant. The rate of increase may be either at a uniform rate as in Fig. 2 or at a nonuniform rate. In computing the creep strain at any time before the load has become constant ($t \lesseqgtr T$) one must include in the summation only those increments which have already become effective, or

$$\epsilon_c = \int_{\tau=0}^{\tau=t} m \frac{t-\tau}{n+(t-\tau)} \frac{d\sigma}{d\tau} d\tau, \quad t \lesseqgtr T \ldots (7)$$

The creep strain at any time after the load has become constant ($t \geqq T$) is obtained by summing up the effects of all load increments (from $\tau = 0$ to $\tau = T$), or

$$\epsilon_c = \int_{\tau=0}^{\tau=T} m \frac{t-\tau}{n+(t-\tau)} \frac{d\sigma}{d\tau} d\tau, \quad t \geqq T \ldots (8)$$

Let it be assumed that, first, the load increases at a uniform rate from $\sigma = 0$ to $\sigma = \sigma_0$, or

$$\frac{d\sigma}{d\tau} = \frac{\sigma}{\tau} = k = \text{constant}, \qquad t < T$$

and second, the concrete is sufficiently aged at the time $\tau = 0$ so that m and n may be considered constant. From Eqs. 7 and 8, one obtains by integration

$$\epsilon_c = km\left[t - n \log\left(1 + \frac{t}{n}\right)\right], \qquad t \lesseqgtr T \ldots (9)$$

$$\epsilon_c = km\left[T + n \log\left(1 - \frac{T}{n+t}\right)\right], t \geqq T \ldots (10)$$

When t becomes infinite, Eq. 10 reduces to

$$\epsilon_c = k\,m\,T \ldots\ldots\ldots\ldots (11)$$

or, since k (the rate of growth of the stress) is the ratio of σ_0 to T,

$$\epsilon_c = m\,\sigma_0$$

giving the same ultimate creep as for a constant sustained load of magnitude σ_0. Figure 2 indicates the creep strain curve B obtained from Eqs. 9 and 10 when the creep constants are $m = 0.40$, $n = 90$ and when the loading data are $\sigma_0 = 300$ psi. compression, $T = 300$ days. Examination of Eq. 11 reveals that the

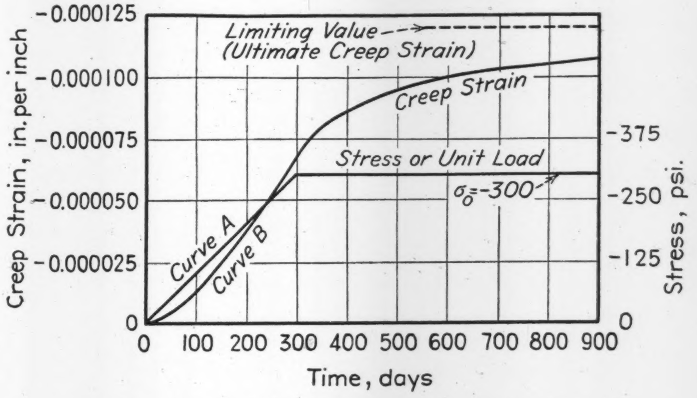

FIG. 2.—Stress and Strain (Uniformly-Increasing Load).

ultimate creep strain for a uniformly increasing stress is independent of the rate of increase of the stress. If the creep constants are variable the creep strain apparently cannot be determined by integration in which case one may compute the creep strain by an ordinary summation process.

Determination of Creep Constants:

By means of the above equations computation of the creep strain curve is possible providing that values of the creep constants are known. In actual practice the order is reversed in that the creep strain curve is given and the problem requires a determination of the creep constants of the concrete. The problem may be briefly stated thus: given, the stress curve (which increases at a uniform rate and finally becomes constant) and the creep strain curve; required, the creep constants m and n. It is assumed that the creep strain curve extends over a time interval sufficiently long to permit a satisfactory determination of the constants. The interval may be either during or after the growth period of the load. As an illustration, a portion of curve B in Fig. 2 will be used as the creep strain curve. The solution given here is dependent upon the following assumptions: (a) the creep strain obeys the laws stated in the equations for continuously applied and uniformly increasing loads, (b) the creep constants are invariable.

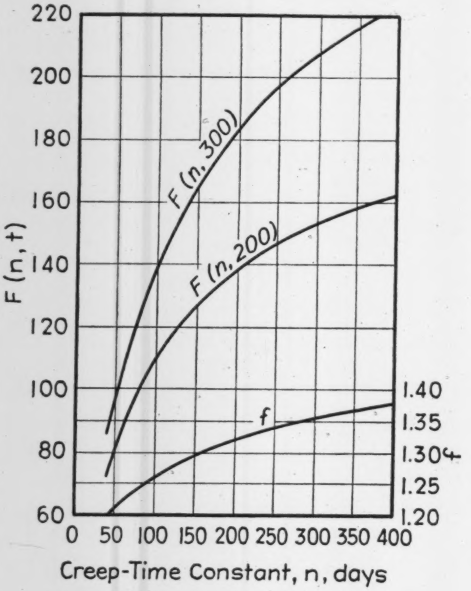

FIG. 3.—Chart for Determining Creep Constants.

Two cases of creep constant determinations are possible. First, when the creep strain curve covers a time interval during the growth period of the load; second, when the creep strain curve covers a time interval after the growth period of the load. In this paper charts and formulas are developed applicable only to the first case. Letting

$$n \log \left(1 + \frac{t}{n}\right) = F(n, t)$$

one has

$$\epsilon_c = km[t - F(n, t)], \qquad t \gtreqless T \ldots (12)$$

by virtue of Eq. 9. Since each term of Eq. 12 is a function of t, one may write

$$\epsilon_c = S(t)$$

where $S(t)$ is defined by Eq. 12. Thus

$$S(t) = km\,[t - F(n, t)], \qquad t \gtreqless T \ldots (13)$$

In Fig. 3 the function $F(n,t)$ is plotted against n for two different values of t, namely, 300 and 200 days. These values of t are not arbitrary but are adapted to the case where the growth period of the load is 300 days. Another function plotted in Fig. 3 is

$$f = \frac{F(n, 300)}{F(n, 200)} \ldots\ldots\ldots\ldots (14)$$

which is obtained by taking the ratios of corresponding ordinates of the first two curves. Note that the function f

is almost constant, varying only from 1.21 (when $n = 50$) to 1.35 (when $n = 300$). For mass concrete in compression it has been found that n is generally between 100 and 200 and a good approximation of f is 1.30. For laboratory concrete in compression n is generally between 20 and 100, a suitable approximation of f being 1.20. Beginning with an approximate value of f one calculates the creep constants by means of the following procedure. Making use of Eq. 13 one may write Eq. 14 in the form

$$f = \frac{S(300) - 300\,km}{S(200) - 200\,km}$$

where k is known from the loading data, where $S(300)$ and $S(200)$ are given by the creep strain curve, where f is known approximately. Solving for the unknown quantity m, one has

$$m = \frac{1}{k} \cdot \frac{S(200)f - S(300)}{(200f - 300)} \ldots\ldots (15)$$

From Eq. 13 one has

$$F(n, 300) = -\frac{1}{km}\,[S(300) - 300\,km] \ldots (16)$$

$$F(n, 200) = -\frac{1}{km}\,[S(200) - 200\,km] \ldots (17)$$

As soon as m has been determined one can calculate $F(n,300)$ or $F(n,200)$ or both. Entering the chart in Fig. 3 with a value of $F(n,300)$ or $F(n,200)$ the value of n is read directly. Having an approximate value of n one can determine the corresponding value of f. If this new value of f differs from the assumed value one should repeat the determination of m and n using the new value of f. Although the process may be repeated any number of times, yielding more and more accurate values of the creep constants, consideration of the practical limitations of strain measurement accuracy seldom warrants going beyond the second approximation.

The above charts and formulas are adapted to the case where the period of growth of the load is 300 days. Nevertheless, the charts and formulas may be readily altered to meet the demands of any other case.

PHYSICAL FACTORS AFFECTING CREEP CONSTANTS

The values of the creep constants are controlled by various physical factors all of which are not of equal importance. As far as is now known, the creep coefficient m depends principally upon the following factors: humidity during loaded period, age at loading, size of specimen, mix proportions, aggregate gradation, type of aggregate, type of cement, and temperature during loaded period. Laboratory studies of the influence of these eight factors generally have been confined to compressive load, consequently little has been learned concerning the relative effects of any one factor in compression and in tension. Based on the gel theory, apparently each of the above factors except *humidity during the loaded period* should produce the same effect under tensile as under compressive load. The effect, upon the creep coefficient, of *humidity during the loaded period* should be opposite in tension and in compression. The variation of the creep-time constant n is slightly erratic and cannot always be interpreted in terms of the various physical factors discussed below.

Humidity During Loaded Period:

The humidity of the atmosphere during the loaded period should affect creep only inasmuch as it influences the expulsion of moisture from the concrete. This is obvious considering that an increase in external humidity reduces

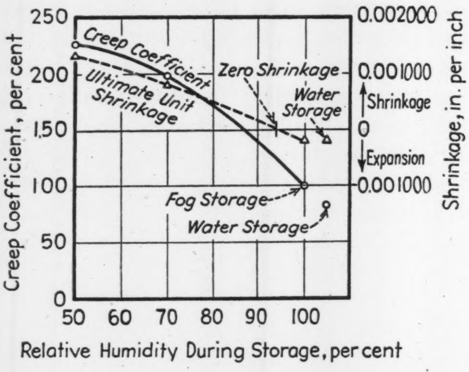

FIG. 4.—Effect of Storage Humidity on Creep and Shrinkage.

Diameter of specimens—4 in.
Aggregate—gravel (Niles, Calif.).
Maximum size of aggregate—unknown.
Combined fineness modulus—5.61.
Cement—normal portland.
Aggregate-cement ratio by weight—5.67.

Water-cement ration by weight—0.59.
Slump—unknown.
Curing—28 days, water, 70 F.
Age at loading—28 days.
Sustained stress—800 psi. compression.
Storage after loading—70 F., humidity indicated.

Source—R. E. Davis, H. E. Davis, and J. S. Hamilton, "Plastic Flow of Concrete Under Sustained Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 360, (Fig. 1, Series 3) (1934).

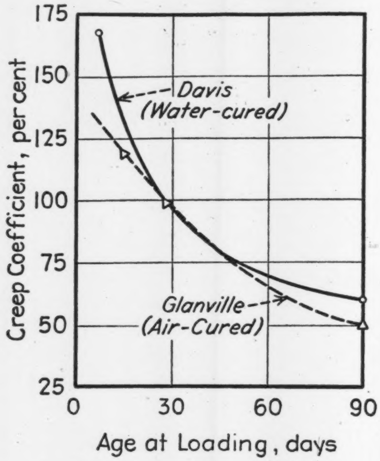

FIG. 5.—Effect of Age at Loading on Creep.

	Investigators	
	Davis	Glanville
Diameter of specimens	4 in.	4 in.
Aggregate	granite (Stevenson Cr. Dam)	Gravel (source unknown)
Maximum size of aggregate	1½ in.	¾ in.
Combined fineness modulus	5.03	5.24
Cement	normal portland	normal portland
Aggregate-cement ratio by weight	5.05	6
Water-cement ratio by weight	0.68	0.70
Slump	3 in.	2 in.
Curing	water, 70 F. until loaded	2 days, 100 per cent relative humidity, 68 F.; then 65 per cent relative humidity, 68 F.
Age at loading	indicated	indicated
Sustained stress	600 psi. compression	600 psi. compression
Storage after loading	water, 70 F.	65 per cent relative humidity, 68 F.

Source—R. E. Davis, H. E. Davis, and J. S. Hamilton, "Plastic Flow of Concrete Under Sustained Stress," *Proceedings*, Am. Soc. Testing Mats. Vol. 34, Part II, p. 370, (Fig 9, Series 2) (1934).
Source—Glanville, *Building Research Technical Paper 12*, p. 16, (Table 3, Batch 30) (1930).

the surface evaporation, increases the resistance to capillary flow, and reduces seepage. The variation of the creep coefficient with relative humidity is shown in Fig. 4. The general effect of atmospheric humidity upon the loss of moisture from concrete may be observed in the shrinkage and swelling of unstressed specimens. Specimens cured in water for 28 days and stored in moist air have been found to shrink considerably when the relative humidity is 70 per cent and to swell slightly when it is 100 per cent. Under the conditions on which Fig. 4 is based, moisture equilibrium for unloaded specimens is obtained at a relative humidity of about 94 per cent.

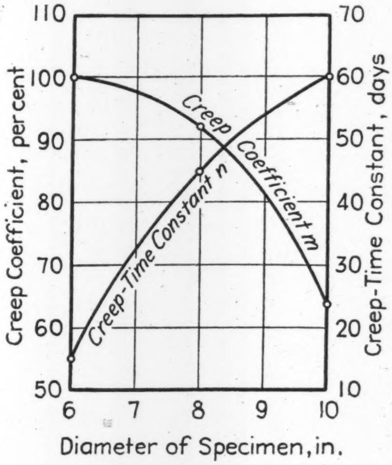

FIG. 6.—Effect of Specimen Size on Creep.

Diameter of specimens—indicated
Aggregate - gravel (source unknown)
Maximum size of aggregate—1½ in.
Combined fineness modulus—5.69
Cement—normal portland
Aggregate-cement ratio by weight—6.95
Water-cement ratio by weight—0.61
Slump—unknown
Curing—28 days, water, 70 F.
Age at loading—28 days
Sustained stress - 800 psi. compression
Storage after loading—100 per cent relative humidity, 70 F.
Source—R. E. Davis, H. E. Davis, and J. S. Hamilton, "Plastic Flow of Concrete Under Sustained Stress," *Proceedings*, Am. Soc. Testing Mats. Vol. 34, Part II, p. 368, (Fig. 7, Series 13) (1934).

Age at Time of Loading:

Creep should depend upon the age of the specimen at the time of load application. The cement hydration has progressed, the less creep should be obtained. The variation of the creep coefficient with the age at loading is shown in Fig. 5. The creep coefficient curve for the water-cured concrete begins flattening earlier than the curve for the air-cured concrete because hydration is achieved more rapidly in the former than in the latter case. A study of creep data for mass-cured concrete[3] (all other conditions being approximately equal to Davis' conditions) discloses that the variation of the creep coefficient with the age at loading closely agrees with Davis' results for water-cured concrete. Without appreciable error, the curve based on Davis' tests may be generally used for water-cured, fog-cured, or mass-cured concretes.

Size of Specimen:

It is generally considered that for cylindrical specimens loaded axially creep varies with the diameter but not with the length of the specimen. As the transverse dimensions increase, the corresponding increase in frictional resistance to flow along the capillary channels results in reduction of seepage. The variation of the creep constants with the diameter is shown in Fig. 6. As the relative humidity is reduced, expulsion of moisture from the concrete to the atmosphere becomes an even more important factor and the effect of the size of specimen on creep may become quite more pronounced. In the transition from small laboratory specimens to massive structures the increase in the dimensions of the concrete body tends to reduce the creep coefficient. In the course of the same transition, however, the maximum size of the aggregate becomes larger. It is shown below that the larger the maximum size of the aggregate, the more creep is obtained. Thus, increase in the dimensions of the specimen and increase in the maximum size of the aggregate are opposing factors.

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[3] Unpublished data obtained in the laboratories of the Bureau of Reclamation, United States Department of the Interior.

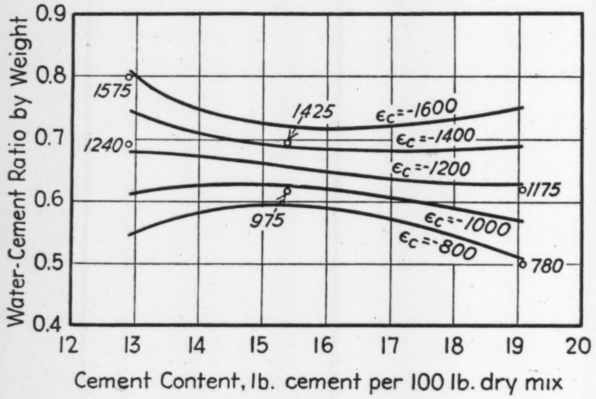

FIG. 7.—Effect of Mix Proportions on Creep.

Diameter of specimens—4 in.
Aggregate—gravel (Berkeley, Calif.)
Maximum size of aggregate - ¾ in.
Combined fineness modulus—4.94
Cement—normal portland
Aggregate-cement ratio -variable (4.25-6.75)
Water-cement ratio—indicated
Slump—unknown
Curing—28 days, 100 per cent relative humidity, 70 F.

Age at loading—28 days
Sustained stress - 800 psi. compression
Storage after loading 50 per cent relative humidity, 70 F.
Source - R. E. Davis, H. E. Davis, and E. H. Brown, "Plastic Flow and Volume Changes of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 321, (Fig. 2, Series 17) (1937).
NOTE.—Creep values measured 600 days after load application.

Mix Proportions:

The proportions of water, cement, and aggregate should have a pronounced effect on the magnitude of creep. The determination of this effect, however, is complicated by the fact that two independent variables are involved. For a given aggregate grading the proportions of the mix can be stated in terms of any two quantities which govern the water content at the time of mixing and the cement content. The two quantities chosen may be: (*a*) aggregate-cement ratio and water-cement ratio, (*b*) cement content and water content, or (*c*) cement content and water-cement ratio. Having selected the two quantities which are to determine the mix, one can represent creep as a function of the mix proportions by contour plotting. Study of available data resulted in Fig. 7 which shows the creep strain plotted against water-cement ratio and cement content. Since only six points were available for determining the contours, Fig. 7 should be considered merely as indicative of the general trend. If data from a large number of mixes were available for comparison, an accurate representation of creep as a function of the mix proportions could be made.

For given curing conditions the proportions of free and adsorbed water at the time of loading depend primarily on the water-cement ratio. The combined quantity of free and adsorbed water at the time of loading, however, chiefly depends on the water content at the time of mixing. From a consideration of the gel theory it is not at once obvious whether the magnitude of creep should be governed by the water-cement ratio or by the water content at the time of mixing or by both. In early tests on the effect of mix proportions on creep neither the water content nor the water-cement ratio were kept constant for a series of tests. The only conclusion that could be drawn was that creep increased as the mix became leaner. Ross (15) suggested that "... creep is increased with leaner mixes only because of increase in water-cement ratio. So long as the mixture is workable ... it seems probable that the aggregate-cement ratio has little effect...." Ross' theory may also be stated thus: within the range of plastic mixes, creep is governed by the water-cement ratio and is practically independent of cement content. If this theory is correct, creep contours plotted against water-cement ratio and cement content should be a series of lines parallel to the axis of cement content. The contours in Fig. 7, in so far as correctly sketched, indicate that: (*a*) Ross' theory holds at least within a limited range of mixes, (*b*) the effect of cement content on creep is always small compared to the effect of water-cement ratio, and (*c*) the creep coefficient rapidly increases with increasing water-cement ratio.

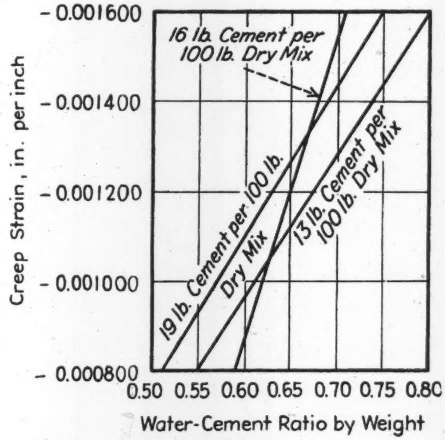

FIG. 8.—Effect of Water-Cement Ratio on Creep (Constant Cement Content).

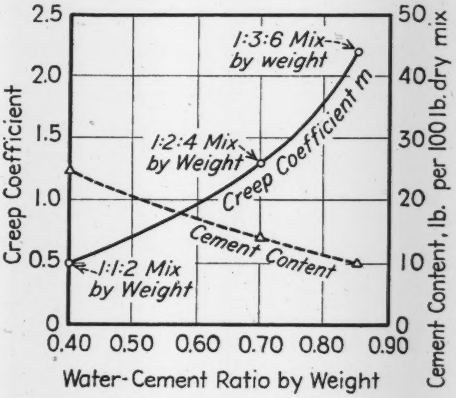

FIG. 9.—Effect of Water-Cement Ratio on Creep (Constant Slump).

Diameter of specimens –4 in.
Aggregate gravel (source unknown)
Maximum size of aggregate –unknown
Combined fineness modulus—5.24
Cement—normal portland
Aggregate-cement ratio by weight—indicated
Water-cement ratio by weight—indicated
Slump—2 in.
Curing—2 days, 100 per cent relative humidity, 68 F.; 26 days, 65 per cent relative humidity, 68 F.
Age at loading – 28 days
Sustained stress 600 psi. compression
Storage after loading—65 per cent relative humidity, 68 F.
Source—Thomas, Building Research Board, Report for 1933, p. 63, Fig. 19, Department of Scientific and Industrial Research, London, England, (1934).

In what follows it will be assumed that for all practical purposes the effect of mix proportions on creep is limited to the effect of the water-cement ratio, not only under the condition of Davis' tests but under all conceivable conditions. The variation of creep with water-cement ratio, for two different test conditions, is shown in Figs. 8 and 9. Figure 8 shows the variation of creep with increasing water-cement ratio

TABLE I.—LABORATORY CREEP DATA.

Cement—normal portland. Curing—28 days at 70 F. Type of loading—compressive. Age at loading, 28 days.

Test Number	Investigator	Source of Data	Investigator's Serial Number	Diameter Cylinder, in.	Length Cylinder, in.	Type and Source of Aggregate	Maximum Size of Aggregate, in.	Fineness Modulus (combined aggregate)	Aggregate-Cement Ratio, by weight	Water-Cement Ratio, by weight	Slump, in.	Length of Curing, days	Humidity Conditions (during curing)	Stress, psi.	Humidity Conditions (during storage)	Storage Temperature, deg. Fahr.	Creep Coefficient, *m*	Creep Constant, *n*, days
1A	Theuer	*Journal of Research*, Nat. Bureau Standards, Vol. 18, No. 2, p. 200 (1937)	24W-27W	3	8	Sand and Gravel (Potomac River)	⅜	3.7[a]	6.37	0.80	Unknown	28	Sealed	434-590	Fog	72	0.08	½
1B			1W-2W	3	8		⅜	4.1[a]	3.00	0.40	Unknown	28	Sealed	1126	Fog	72	0.07	½
2A	Davis	A.S.T.M., *Proceedings*, Vol. 34, Part II, p. 381, Fig. 17 (1934)	8	5	14	Sand and Gravel (Berkeley, Calif.)	½	4.47	4.80	0.53	6	21 7	Fog Water	935	Water	70	0.35	100
2B			8	5	14		½	4.47	8.40	0.83	6	21 7	Fog Water	558	Water	70	0.54	120
3	Davis	A.S.T.M., *Proceedings*, Vol. 34, Part II, p. 364, Fig. 4 (1934)	16	4	10	Sand and Gravel (Source Unknown)	Unknown	4.83	6.65	0.75	Unknown	27 1	Fog Water	800	Water	70	0.36	25
4	Davis	A.S.T.M., *Proceedings*, Vol. 34, Part II, p. 370, Fig. 9 (1934)	2	4	14	Sand and Granite (Stevenson Creek Dam)	1½	5.03	5.05	0.68	3	28	Fog	600	Water	70	0.68	160
5A	Davis	A.S.T.M., *Proceedings*, Vol. 34, Part II, p. 365, Fig. 5 (1934)	13	6	12	Sand and Gravel (Source Unknown)	¾	5.15	6.95	0.61	Unknown	28	Fog	800	Fog	70	0.28	15
5B	Davis	A.S.T.M., *Proceedings*, Vol. 34, Part II, p. 368, Fig. 7 (1934)	13	6	12		1½	5.69	6.95	0.61	Unknown	28	Fog	800	Fog	70	0.36	15
5C			13	8	16		1½	5.69	6.95	0.61	Unknown	28	Fog	800	Fog	70	0.33	45
5D			13	10	20		1½	5.69	6.95	0.61	Unknown	28	Fog	800	Fog	70	0.23	60
6A	Davis	A.C.I., *Proceedings*, Vol. 27, p. 855, Fig. 5 (1931)	7	6	12	Sand (Monterey, Calif.) and Gravel (Niles, Calif.)	Unknown	5.55	7.7	0.65	3 to 4	7 21	Fog Water	800	Water	70	0.24	25
6B			7	6	12		Unknown	5.70	4.4	0.42	3 to 4	7 21	Fog Water	800	Water	70	0.22	35
7	Davis	A.S.T.M., *Proceedings*, Vol. 34, Part II, p. 360, Fig. 1 (1934)	3	4	14	Sand and Gravel (Niles, Calif.)	Unknown	5.61	5.67	0.59	Unknown	28	Fog	800	Fog	70	0.46	140
8A	Crepps	Purdue U. Eng. Exp. Sta. *Bulletin*, Vol. XVIII, No. 5, p. 32, Fig. 21 (1934)	Speed	6	24	Sand (Elgin, Ill.) and Limestone (Greencastle, Ind.)	1½	5.7	6.40	0.53	1	28	Fog	500	Fog	70	0.32	75
8B			Speed	6	24		1½	5.7	6.94	0.53	2¼	28	Fog	500	Fog	70	0.35	55

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[a] Estimated value.

when the cement content is constant. The data were obtained by taking profiles across the contours in Fig. 7. Although the contouring in Fig. 7 may not be precise, the general trend of the curves in Fig. 8 is believed correct. Figure 9 indicates the variation of creep with water-cement ratio when the slump is constant. For the conditions of Thomas' tests the creep coefficient is very nearly proportional to the square of the water-cement ratio.

Aggregate Gradation:

It is believed that creep to a large measure depends upon the gradation of the aggregate. However, no satisfactory investigation along this line is known to have been undertaken. A survey of available creep data discloses that the creep coefficient increases with the fineness modulus of the aggregate. In general, the fineness modulus depends on both the maximum size of the aggregate and the quality of the grading. For well-graded aggregates, the fineness modulus is a rough measure of, and increases with, the maximum size of aggregate. The degree of variation of the creep coefficient with the fineness modulus may be derived from Table I, containing creep data for 15 concretes. These concretes differ in type of aggregate, in aggregate gradation, and in mix proportions. Comparison of creep coefficients for mixes which differ in water-cement ratio and in aggregate gradation (tests 1A and 1B, 6A and 6B) reveals that the effect of an increase in fineness modulus offsets the effect of a decrease in water-cement ratio. Refer to Fig. 9 and to Table I (tests 2A and 2B) for a concept of the effect of water-cement ratio on the creep coefficient. For mixes which differ only in aggregate gradation (tests 5A and 5B) an increase in fineness modulus results in an increase in creep coefficient.

Under properly controlled laboratory conditions, as the maximum size of the aggregate increases, the fineness modulus becomes greater and the dimensions of the specimen are increased. Thus in the transition from laboratory concrete to mass concrete it is possible to roughly correlate two opposite effects which occur simultaneously: (*a*) the increasing dimensions of the specimen tend to reduce the creep coefficient, (*b*) the increasing fineness modulus tends to increase the creep coefficient. The combined effect of increasing fineness modulus and increasing dimensions of the specimen may be observed by comparing the creep coefficients of the laboratory concretes listed in Table I (tests 2A, 5A, 5B, 5C, 5D, 7, 8A, 8B) and the mass concrete in Boulder Dam, block J4, elevation 595. The fineness moduli of the aggregates used in the laboratory mixes ranged from 4.5 to 5.7. The fineness modulus of the Boulder Dam aggregate was 7.0. Other conditions for the laboratory mixes and the Boulder mix were roughly equal, as shown by the following data. Laboratory concrete: water-cement ratio, 0.53 to 0.61; cement, normal portland; curing and storage, at 70 F. in fog or water; compressive load applied at the age of 28 days. Boulder Dam concrete: water-cement ratio, 0.54; cement, normal portland; mass curing, starting temperature 80 F. and an average temperature of approximately 68 F. during the first 300 days; compressive load applied in increments beginning at the age of 5 days. The creep coefficient of the laboratory concretes ranged from 0.2 to 0.4 whereas the creep coefficient of the Boulder Dam concrete, as calculated from the records of embedded strain meters, was 0.6. The comparison demonstrates that in the transition from laboratory specimens to massive structures the effect of the increasing fineness modulus overshadows the effect of the increasing dimensions of the specimen.

Type of Aggregate:

Tests by Davis (4) indicate that concretes having the same mix proportions and the same aggregate grading but fabricated with various aggregates have different creep coefficients. Figure 10 presents the results of a test in which five crushed-rock aggregates and one natural aggregate were used. Each crushed-rock aggregate was combined so as to give exactly the same gradation as the natural aggregate (Niles sand and gravel). All six concretes had the same mix proportions. A comparison of the five crushed-rock concretes shows that there is a considerable range of variation in the magnitude of the creep coefficient and that the creep-time constant is practically the same for all five crushed-rock concretes.

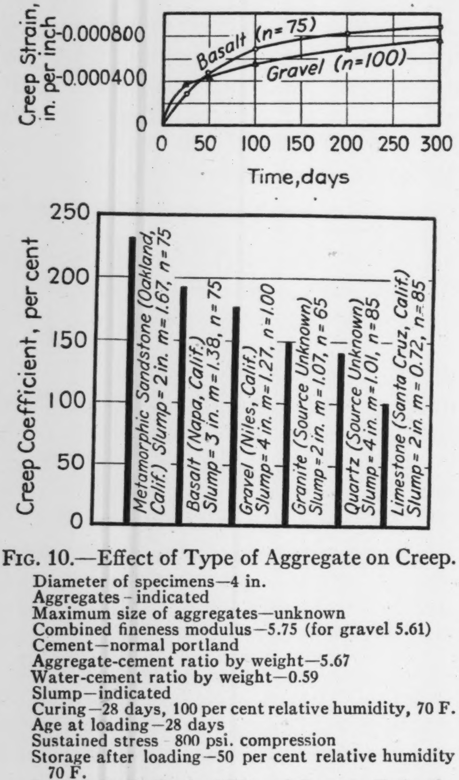

FIG. 10.—Effect of Type of Aggregate on Creep.

Diameter of specimens—4 in.
Aggregates—indicated
Maximum size of aggregates—unknown
Combined fineness modulus—5.75 (for gravel 5.61)
Cement—normal portland
Aggregate-cement ratio by weight—5.67
Water-cement ratio by weight—0.59
Slump—indicated
Curing—28 days, 100 per cent relative humidity, 70 F.
Age at loading—28 days
Sustained stress—800 psi. compression
Storage after loading—50 per cent relative humidity 70 F.
Source—R. E. Davis, "Flow of Concrete Under the Action of Sustained Loads," *Proceedings*, Am. Concrete Inst., Vol. 27, p. 857, (Fig. 6, Series 4) (1931).

The following aggregate characteristics possibly may influence the variation of the creep constants for concretes made with different aggregates: (*a*) mineral composition, (*b*) particle shape, (*c*) surface texture and pore structure.

(*a*) It is possible that the creep of concrete is caused not only by yielding of the gel but also by crystalline slip. The magnitude of crystalline slip should be expected to be different for aggregates of different mineral composition. Variations of the creep coefficient, therefore, may be partly due to variations in crystalline slip. It appears, however, that crystalline slip alone cannot account for the entire amount of variation shown in Fig. 10.

(*b*) The ideal gradation of an aggregate depends upon the shape of the particles. Hence for a given aggregate gradation and given mix proportions, the shape of particles should influence all important properties of concrete.

(*c*) In regard to the gel theory the size, shape, and structure of the pore spaces and capillary channels in the aggregate possibly have considerable bearing upon the creep constants. The pores near the surface of the particles are especially important. This is obvious when one considers, by analogy, that the strength of bond between the aggregate particles and the surrounding matrix is dependent upon mechanical anchorage sometimes due to roughness of the aggregate and more often due to adhesion caused by suction of the paste into superficial pores in the aggregate.

Type of Cement:

A direct comparison of the creep in low-heat cement and in normal cement

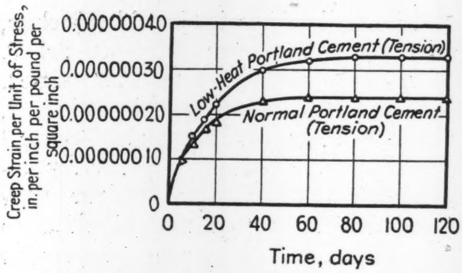

FIG. 11.—Effect of Type of Cement on Creep.

Diameter of specimens—6 in.
Aggregate—gravel (Columbia River)
Maximum size of aggregate—1½ in.
Combined fineness modulus—5.46
Cement—indicated
Aggregate-cement ratio by weight—9 (estimated)
Water-cement ratio by weight {0.69 for normal portland cement; 0.66 for low-heat portland cement}
Slump—1 in.
Curing—Mass, beginning at 40 F.
Age at loading—28 days
Sustained stress {for normal: 200 psi. compression; 50 psi. tension; for low-heat: 400 psi. compression; 100 psi. tension}
Storage after loading—80 F. constant
Source—R. E. Davis, H. E. Davis, and E. H. Brown, "Plastic Flow and Volume Changes of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 322, (Fig. 3, Series 18) (1937).

Type of Loading	Type of Cement	*m*	*n*
Compression	Low-heat portland	0.25	20
	Normal portland	0.19	35
Tension	Low-heat portland	0.35	6
	Normal portland	0.26	8

concretes is shown in Fig. 11 and indicates that: (*a*) in either tension or compression, the creep coefficient for concrete made with low-heat cement is roughly one-third greater than for concrete made with normal cement; (*b*) the magnitude of the creep-time constant does not appear to be materially affected by the type of cement. The comparison serves to explain why low-heat cement concrete possesses greater stress-relieving properties which result in superior resistance to cracking.

Temperature During Loaded Period:

Creep should be influenced by both the temperature of the concrete and the temperature of the surrounding atmosphere. The concrete temperature affects the physical properties of the gel and of the water which seeps through the capillary passages. As the temperature increases the viscosity of the water is reduced and seepage is facilitated. For a given relative humidity the rate of evaporation increases with increasing air temperature. A higher rate of

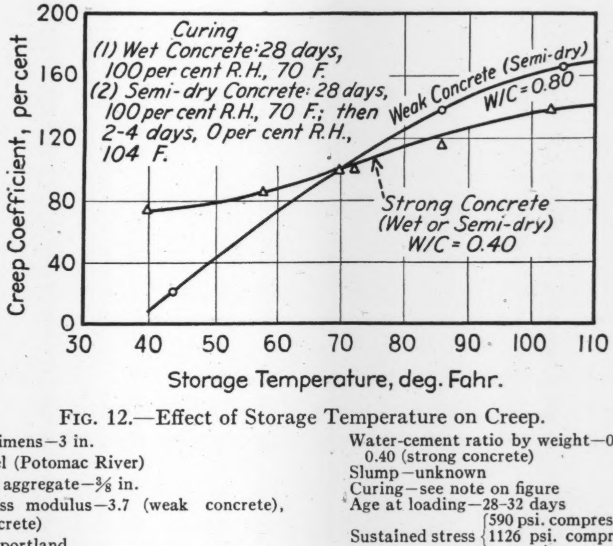

FIG. 12.—Effect of Storage Temperature on Creep.

Diameter of specimens—3 in.
Aggregate—gravel (Potomac River)
Maximum size of aggregate—⅜ in.
Combined fineness modulus—3.7 (weak concrete), 4.1 (strong concrete)
Cement—normal portland
Aggregate-cement ratio by weight—6.37 (weak concrete), 3.00 (strong concrete)
Water-cement ratio by weight—0.80 (weak concrete), 0.40 (strong concrete)
Slump—unknown
Curing—see note on figure
Age at loading—28–32 days
Sustained stress {590 psi. compression (weak concrete); 1126 psi. compression (strong concrete)}
Storage after loading—sealed in rubber, temperature variable

Source—A. U. Theuer, "Effect of Temperature on the Stress-Deformation of Concrete," *Journal of Research*, Nat. Bureau Standards, Vol. 18, No. 2, p. 200 (1937).

evaporation results in greater loss of moisture from the surface of a loaded concrete specimen and consequently results in greater creep. Thus air temperature becomes an important consideration in the analysis of creep phenomena in laboratory specimens.

Theuer (18), having eliminated evaporation by sealing his test specimens in rubber jackets, demonstrated that creep varies appreciably with the temperature of the concrete. In Theuer's tests two concrete mixes of approximately the same consistency but of different cement content were employed. The following curing procedure was applied to each mix: all specimens were fog-cured for 28 days at which age they were divided into two groups, one of which was immediately sealed in rubber jackets, and the other oven dried at 104 F. from 2 to 4 days and then sealed. Theuer's results, shown in Fig. 12, indicate that: (*a*) the higher the gel temperature, the higher the creep coefficient; (*b*) for mixes of the same consistency, the variation of the creep coefficient; for a given temperature change is greater when the water-cement ratio is high than when it is low, (*c*) the quantity of free and adsorbed water present in the concrete at the time of application of the load has no appreciable effect on the law of variation of creep with temperature.

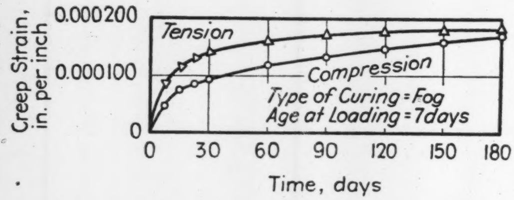

FIG. 13.—Creep Under Compressive and Tensile Load.

	Fog-Cured	Mass-Cure
Diameter of specimens	6 in.	6 in.
Aggregate	gravel (Berkeley, Calif.)	gravel (Columbia River)
Maximum size of aggregate	¾ in.	1½ in.
Combined fineness modulus	4.57	5.46
Cement	normal portland	normal portland
Aggregate-cement ratio by weight	6.00	9 (estimated)
Water-cement ratio by weight	0.63	0.69
Slump	3 in.	1 in.
Curing	7 days and 28 days, 100 per cent relative humidity, 70 F.	Mass, beginning at 40 F.
Age at loading	(1) 7 days, (2) 28 days	28 days
Sustained stress (1)	100 psi compression and tension	200 psi. compression
Sustained stress (2)	150 psi. compression and tension	50 psi. tension
Storage after loading	50 per cent relative humidity, 70 F.	80 F.

Source—R. E. Davis, H. E. Davis, and E. H. Brown, "Plastic Flow and Volume Changes of Concrete," *Proceedings*, Am. Soc. Testing Mats. Vol. 37, Part II, p. 323, (Fig. 4, Series 20); also, p. 322, (Fig. 3, Series 18) (1937).

Type of Curing	Nature of Stress	*m*	*n*
Mass curing for 28 days	Compression	0.19	35
	Tension	0.26	8
Fog curing for 28 days	Compression	1.94	45
	Tension	2.17	20
Fog curing for 7 days	Compression	2.07	50
	Tension	1.97	15

Nature of the Load:

Several tests by Davis (6) indicate

that creep in tension is characterized by a relatively high time-rate immediately after the application of the load, that is, the creep-time constant (the time required to obtain 50 per cent of the ultimate creep) is considerably less for tensile than for compressive load. The results of three tests comparing creep in tension and in compression are given in Fig. 13. The creep-time constants, in so far as the average values based on these tests are accurate, for identical concrete specimens under tensile and under compressive load, respectively, are in the ratio of one to three. The creep coefficients are of the same order of magnitude under both tensile and compressive load.

Humidity Prior to Loading:

The influence, upon creep, of the relative humidity of the atmosphere to which a concrete specimen is exposed prior to loading, was disclosed by Dutron (8, 9). In what follows, the term *curing period* will be understood to imply the time from the casting of the specimen to the application of the load. Conditions existing prior to and during the loaded period will be referred to, respectively, as *curing* and *storage* conditions. Dutron tested two groups of concrete prisms, 10 by 10 cm. in cross-section, which had been cured at 70 F. for 60 days as follows: one group in water, the other group in air of 50 per cent relative humidity. Some members of each group were loaded in water (70 F.), others in air (70 F.) of 50 per cent relative humidity. The relative magnitudes of ultimate creep for different combinations of curing and storage conditions were as follows: water-cured, water-stored, 100 per cent; air-cured, water-stored, 170 to 220 per cent; water-cured, air-stored, 400 to 450 per cent; air-cured, air-stored, 310 to 340 per cent. It will be noted that air-cured specimens showed either more or less creep than water-cured specimens stored under the same conditions, depending on whether storage was in water or in air. In order to understand this apparently peculiar behavior, it is necessary to recognize the existence of two separate phenomena which occur during the curing period: (*a*) hydration of the cement, (*b*) shrinkage or swelling of the concrete. The effect, upon creep, of humidity conditions prior to loading is the sum of the effects of these two phenomena.

(*a*) It has been shown that the tendency of concrete to creep decreases as more cement hydration is obtained. Thus, if two identical specimens are cured under the same conditions and one is loaded at an earlier age than the other, the former will tend toward greater ultimate creep strain than the latter. Similarly, if two identical specimens are cured for the same length of time, one in dry air and one in water, the former should have a greater tendency to creep than the latter since during the same time-interval less cement hydration occurs in air-stored than in water-stored concrete.

(*b*) Air-cured concrete shrinks and water-cured concrete swells during the curing period. Under compressive load, pre-swelled specimens are capable of more creep than pre-shrunk specimens. The difference in the creep of pre-swelled and pre-shrunk specimens is more pronounced the greater the ability of the pre-swelled concrete to expel moisture into the atmosphere. Conditions which facilitate the expulsion of moisture are: small cross-section of the specimen, storage in dry air, high ambient temperature during the loaded period.

Thus the effect of hydration and the effect of pre-shrinkage or pre-swelling are opposing factors; the former tends to produce less creep in water-cured than in air-cured concrete, whereas the latter tends to produce more creep in water-cured than in air-cured concrete. In the tests made by Dutron, the net result of the two opposing factors was: for concrete loaded in water, air-cured specimens exhibited from 70 to 120 per cent more creep than water-cured specimens; for concrete loaded in air, air-cured specimens showed about 25 per cent less creep than water-cured specimens. Dutron's results are explained by the following. Storage in water minimizes the effect of pre-swelling on creep thus permitting the effect of hydration to obscure the former effect. On the contrary, for concrete loaded in air the effect of pre-swelling is sufficient to offset the effect of hydration. It should be borne in mind that Dutron's specimens were of small cross-section. As the transverse dimensions of a specimen increase, the expulsion of water becomes more difficult and the effect of pre-swelling is lessened. In a sufficiently large specimen the effect of hydration always governs irrespective of storage conditions.

Degree of Compaction:

From the viewpoint of the gel theory it is conceivable that creep should be influenced by the degree of compaction of the concrete mass. A high degree of compaction should result in less seepage with a consequent reduction in creep. The effect of vibration-compacting on the creep properties of concrete has been investigated by Davis (7) and by Thomas (20). Unfortunately, no complete test data are available. Davis found that the creep of vibrated concrete is less than that of concrete not so treated. Thomas' results reveal that no real difference exists between the creep coefficients of hand-compacted and vibration-compacted concretes of identical water-cement ratios. Thus the relative significance of this factor is obviously dependent upon future experimental findings.

Temperature Prior to Loading:

Extensive test results are necessary in order to establish the effect, on creep constants, of the temperature of the concrete prior to loading. Laboratory tests dealing with the effect of this factor are now in progress in the laboratories of the Bureau. The author is of the opinion that high temperature of the concrete during the curing period, unaccompanied by moisture loss from the concrete, should accelerate cement gel formation. Under such humidity conditions, rise in curing temperature should tend to increase the magnitude of creep.

SUMMARY

The theory as presented in this paper provides a provisional picture of concrete creep until such time as confirmed by comprehensive experimental observations. The existing laboratory data are as yet too incomplete to allow a more precise indication of the actual relations existing between the creep constants and the various physical factors. However, it is quite likely that new experiments along the lines indicated in this paper will lead to confirmation of these relations.

1. Creep strain is proportional to stress, accurately for stresses from 0 to 650 psi. and approximately for stresses from 650 to 1500 psi., and is defined by

$$\epsilon_c = f(t) \cdot \sigma$$

2. For constant sustained stress the creep-time relation is expressed by

$$\epsilon_c = \frac{mt}{n + t} \cdot \sigma$$

3. For uniformly increasing sustained stress, at any time before the stress becomes constant

$$\epsilon_c = km\left[t - n\log\left(1 + \frac{t}{n}\right)\right], \qquad t \leqq T$$

and at any time after the stress has become constant

$$\epsilon_c = km\left[T + n\log\left(1 - \frac{T}{n+t}\right)\right], \qquad t \geqq T$$

The magnitude of creep is governed by the following physical factors in so far as they affect the creep coefficient m and the creep-time constant n.

4. Humidity of the atmosphere during the loaded period affects creep only inasmuch as it influences the expulsion of moisture from the concrete. High percentage relative humidity during the loaded period results in a low creep coefficient. Moisture equilibrium for unloaded specimens is obtained at a relative humidity of about 94 per cent.

5. Decrease in the creep coefficient is effected by increase in age at loading. For water-cured, fog-cured, or mass-cured concrete the variation of the creep coefficient with age at loading is small for ages greater than 90 days.

6. Increasing the diameter of the specimen results in a decrease in the creep coefficient with a corresponding rise in the creep-time constant.

7. Creep is governed mainly by the water-cement ratio of the concrete and by the fineness modulus of the aggregate. Within a limited range of mixes the creep coefficient rapidly increases with increasing water-cement ratio and is practically independent of the cement content of the mix. The creep coefficient increases with increasing fineness modulus of the aggregate.

8. In the transition from laboratory concrete to mass concrete the effect of the increasing dimensions is obscured by the effect of the increasing fineness modulus.

9. The magnitudes of the creep constants depend upon the mineral composition, particle shape, and surface texture of the aggregate.

10. In either compression or tension, the creep coefficient of concrete made with low-heat cement is about one-third greater than that of normal cement concrete. The magnitude of the creep-time constant does not appear to be materially affected by the type of cement.

11. The higher the temperature of the cement gel, the higher the creep coefficient. For mixes of the same consistency, the variation of the creep coefficient for a given temperature change is greater when the water-cement ratio is high than when it is low. The quantity of free and adsorbed water present in the concrete at the time of application of the load has no appreciable effect on the law of variation of creep with temperature.

12. The creep-time constant of concrete under tensile load is roughly one-third of that of an identical concrete under compressive load. The creep coefficient generally is of the same order of magnitude under both compressive and tensile load.

13. The importance of humidity conditions prior to loading is dependent upon the net effect of two opposing phenomena: cement hydration and shrinkage or swelling of the concrete.

Acknowledgment:

The author wishes to acknowledge the valuable assistance of D. O. Ehrenburg, Assistant Engineer, with whom the author collaborated in an investigation of the volume changes in mass concrete. The investigation, conducted in the Dams Division of the Bureau of Reclamation, United States Department of the

Interior, is under the immediate supervision of C. Rawhouser, Associate Engineer. All work in the Division of Dams is under the direction of K. B. Keener, Designing Engineer in charge of dams. All design and research work of the Bureau of Reclamation is under the direction of J. L. Savage, Chief Designing Engineer. All engineering and construction is under the general direction of R. F. Walter, Chief Engineer, with headquarters in Denver, Colo. All activities of the Bureau are under the general charge of John C. Page, Commissioner, with administrative offices in Washington, D. C.

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AUTOGENOUS VOLUME CHANGES OF CONCRETE

By Harmer E. Davis[1]

Synopsis

Autogenous volume changes of concrete are defined as those which are due primarily to volume changes of the cement paste as the result of hydration of the cement and are differentiated from the effects of moisture movements to or from the atmosphere, of temperature variations, and of load. In connection with strain measurements in mass-concrete structures extending over periods of several years, volume changes of this character become large enough to require consideration in the interpretation of results. Tests extending over a period of 5 yr. indicate that under certain conditions the autogenous shrinkage may be of the order of 100 millionths. Data on autogenous volume changes are also of importance in connection with the general problem of the hydration process in cement. Data reported include results of tests on concretes made with normal, low-heat and portland-pozzuolana cements.

Autogenous volume changes of concrete are defined as those which result from alterations in physical and chemical structure within the mass itself, due to causes other than: (1) movement of moisture to or from the surrounding atmosphere, (2) rise or fall in temperature, and (3) stresses caused by external load or restraint. If the effect of expansion or contraction due directly to variations in temperature is eliminated, autogenous volume changes are those which would be determined from an unrestrained mass of concrete sealed against gain or loss of moisture. They have sometimes been referred to as "chemical" volume changes. The primary causes of autogenous volume changes are considered to be the volumetric changes in the crystalline constituents of cement during the process of hydration and in the gelatinous and crystalline products of hydration.

In measurements of strains in massive concrete structures, such as dams, and in tests of mass concrete involving the use of sealed specimens, autogenous movements may require consideration in the interpretation of results, especially if measurements are continued over a number of years. Further, information on autogenous volume changes have bearing upon the problem of the nature of the hydration process in cement and hence upon the problem of durability of concrete.

Practically all measurement of thermal and hygral volume changes include the effects of autogenous volume changes. However, it would ordinarily be neither practical nor desirable to attempt to differentiate between the autogenous movements, which are believed usually to be of relatively small magnitude, and the direct effects of drying and temperature change.

The expansions of "unsound" cements,

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due to hydration of excessive free lime or of magnesia are autogenous in character. Also, the hydration of minerals of certain types of aggregate may produce expansions of an autogenous nature. However, behavior of this sort is not within the scope of this paper.

The concept of autogenous volume change is not new. Lynam[2] makes reference to it, although he points out that at that time (1934) there appeared to be no records of long-time experiments relating to autogenous shrinkage alone. Based on the collodial theory of hardening, Kühl[3] and Lea and Desch[4] discuss internal changes that take place as hydration proceeds. The shrinkage of cement paste during setting has been a matter of record for many years; White[5] refers to experiments by Le Chatelier as early as 1900. Pertinent dilatometer experiments made more recently on the volume changes occurring during setting and shortly thereafter have been reported by Neville and Jones,[6] by Powers,[7] and by Hemeon.[8] Volume changes up to the age of 90 days, determined by the U. S. Bureau of Reclamation at Denver from tests on sealed, adiabatically-cured concrete specimens containing portland and portland-pumicite cements, after thermal volume changes were deducted, are reported by Savage.[9]

MECHANISM OF AUTOGENOUS VOLUME CHANGE

While the details are incomplete, and admittedly inaccuracies may exist, such evidence as is available favors the following general view of the mechanism of autogenous volume change in relation to the hydration process:

Owing to the formation of products which have lesser volume than the sum of the volumes of the original components taken separately, the system cement plus water contracts as hydration proceeds. Although the volume of the total system, cement plus water, may decrease, the absolute volume of solids increases, since the newly formed solids are the result of chemical combination of the original solids and water.

A mixture of cement and water placed in a flask, with sufficient clear water added to extend into the neck of the flask to act as a volume indicator, shows continuous decrease in total volume if temperature changes are eliminated, and the shrinkage continues even after the paste has hardened. All of the principal compounds of portland cement appear thus to "absorb" water. Powers[7] found after 28 days, absorptions in terms of percentage by weight of the compound approximately as follows: 2CS, 1 per cent; 3CS, 5 per cent; 3CA, 17 per cent; 4CAF, 9 per cent. A volume decrease also accompanies the formation of calcium hydroxide from lime and water. These changes in volume are attributable in part to the formation of hydrated compounds and in part to the strong adsorption of moisture films by the gel products.

In a mass of paste or concrete, after the paste has hardened, the solids form a continuous structure or network, interpersed throughout with pores or channels which may contain more or less free water. It is thus possible for the hardened solid mass to expand, even though the total volume of cement and water is decreasing, due to the use of internally-contained free water in the formation of new solid hydration products and in expansion of the gel. Thus, in an experiment with cement paste in a flask such as referred to above, the hardened paste may expand sufficiently to break the flask, even though the water level is decreasing.

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[2] C. G. Lynam, "Growth and Movement in Portland Cement Concrete," p. 26, Oxford University Press, London (1934).

[3] H. Kühl, "Cement Chemistry in Theory and Practice," p. 36, Translation by J. W. Christelow, Concrete Publications, Ltd., London (1931).

[4] F. M. Lea and C. H. Desch, "The Chemistry of Cement and Concrete," p. 175, Longmans, Green and Co., London (1935).

[5] A. H. White, "Volume Changes in Hydrated Portland Cement," *Concrete*, Vol. 33, No. 2, August, 1928, pp. 41–45.

[6] H. A. Neville and H. C. Jones, "Volume Changes During Hydration of Gelatin, Cement, and Plaster of Paris," *Colloid Symposium Monograph*, Vol. 6, pp. 309–318 (1928).

[7] T. C. Powers, "The Absorption of Water by Portland Cement During the Hardening Process," *Industrial and Engineering Chemistry*, Vol. 27, No. 7, July, 1935, pp. 790–794.

[8] W. C. L. Hemeon, "Setting of Portland Cement—Rates of Contraction and Heat Evolution," *Industrial and Engineering Chemistry*, Vol. 27, No. 6, June, 1935, pp. 694–699.

[9] J. L. Savage, "Special Cements for Mass Concrete," p. 90 (Prepared for the Second Congress of the International Commission on Large Dams), Denver, Colo. (1936).

At later ages, in a mass sealed against transfer of moisture to or from the surroundings, continuation of hydration may utilize not only all the free water available in the pores but also may remove some of the more loosely held water from the gel. Loss of water by the gel results in a tendency for the gel structure to contract; thus under conditions such as these, a concrete mass would undergo decrease in volume.

Whether a hardened concrete will expand or contract would appear, then, to depend upon the relative importance of two competing factors: (1) production of new gel and consequent expansion thereof by absorption of free pore water, and (2) shrinkage of gel caused by extraction of water therefrom for reaction with previously unhydrated cement. Results of tests, shown later, indicate that the latter is the dominant factor at the later ages. In so far as the paste is concerned, the following would appear to be the major variables which influence the behavior of a particular concrete: (1) composition of the cement (affecting the nature and rate of chemical reactions and type of reaction products), (2) amount of original mixing water (affecting the rate of early reactions, the porosity of the paste, and the later availability of free pore water), (3) curing conditions (affecting the rate of reaction), and (4) time (affecting the extent of reaction).

VOLUME CHANGES DURING THE SETTING PERIOD

It is a well-known fact that, up to the time of set, fresh concrete settles in the forms with an appreciable decrease in volume. This volume decrease is herein called *subsidence* to distinguish it from drying shrinkage. The most important factor in subsidence is ordinarily the bleeding of free water to the top of the mass with attendant settlement of the solids. The major portion of the bleeding action usually occurs within an hour or so after the concrete has been placed.[10] Under job conditions, much of the bled water may be lost by drainage from the forms or by evaporation. While total subsidence during setting may in extreme cases amount to 1 per cent or more of the volume, the volume change due to hydration and adsorption effects appears to be of the order of 0.1 per cent with ordinary cements.

While the true autogenous shrinkage, usually small compared with subsidence due to bleeding, can be considered to be without practical significance during the early ages, it probably contributes to the occurrence of certain types of small checks and cracks that are seen on formed surfaces after form removal. For example, with some rapid-hardening cements, small vertical cracks are sometimes observed to occur along vertical stirrups in beams, where the stirrups are close to the forms, even though the forms are tight and nonabsorbent. Although it is a matter of conjecture it seems likely also that during the period after set has taken place, but while the paste is still relatively soft and weak, similar action would tend to cause the formation of small internal cracks in the paste between aggregate particles in concretes of very dry consistency.

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[10] T. C. Powers, "The Bleeding of Portland Cement Paste, Mortar and Concrete," *Bulletin No. 2*, Research Laboratory of Portland Cement Assn. (1939).

SECONDARY EFFECTS OF THERMAL CHANGES

Certain hysteresis-like secondary effects of volume changes due to temperature may be considered to be autogenous in character. Movements of this type are important in relation to measurements of autogeneous volume changes of the chemical type because in all known attempts to measure volume changes of sealed cement or concrete specimens some thermal changes have been involved and corrections presumably have been made for them.

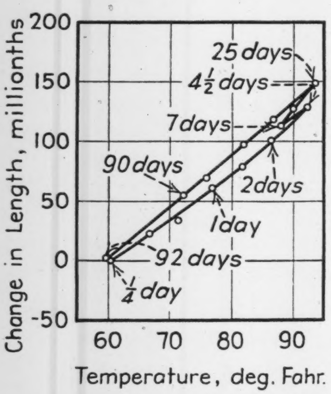

FIG. 1.—Change in Length *versus* Temperature, Mass Curing.

NOTES:

Specimens, 12-in. concrete cubes sealed in thin copper containers immediately after casting. Mass curing from initial temperature of 57 F. to age 3 months. Strains measured by Carlson strain meters embedded parallel to original free surface at midheight. Portland-pozzuolana cement, 25 per cent calcined, ground volcanic sand, 75 per cent portland cement of computed composition as follows: 3CS, 52 per cent; 3CA, 5 per cent. Specific surface, 2000 sq.cm. per g. (Klein basis). Aggregate, 0 to 3-in. gravel, fineness modulus 6.31. Cement content 1.0 bbl. per cu. yd. Slump 3 in., water-cement ratio 0.63 by weight. Values are averages for 2 specimens.

An example of volume change which involves the secondary results of thermal movements is shown in Fig. 1. In this case, sealed specimens were carried through a temperature cycle corresponding to that existing within the mass of a large dam. During the period of falling temperature, there was a residual expansion or growth of the concrete with respect to lengths observed during the period of rising temperature. The maximum difference in length at any one temperature was about 10 millionths. While differential movements of the cement paste and the aggregate, caused by unequal expansions of these two constituents of the concrete, probably played a part in the hysteresis-like nature of the strain cycle, it is not unlikely that the results were appreciably affected by volume changes of chemical origin since the tests were made during the period when hydration of the cement was proceeding rapidly.

AUTOGENOUS VOLUME CHANGE OF HARDENED CONCRETE

In the determination of volume changes of hardened concrete, it would be desirable (theoretically at least) to trace the course of volume change from the time of casting, or at least from time of set. Actually, however, specimens and most methods of measurement suitable for use during the setting period are generally not satisfactory for determining length changes of hardened concrete over long periods. Further, the time of set of a concrete mass does not appear to be a definite or significant point as far as volume changes are concerned; also, for some time after what is considered as set has occurred, concrete remains too soft to permit the use of the usual type of strain gage or strain meter. Hence, practically, base measurements are arbitrarily made at ages of one, two, or even three days. In so far as autogenous volume changes over long periods are concerned, some variation in the base age does not appear to be of much importance.

TABLE I.—LENGTH CHANGES OF SEALED CONCRETE SPECIMENS.

Cement		Cement Content, bbl. per cu. yd.	Water-Cement Ratio, by weight	Change in Length, millionths[b]			
Type	Specific Surface, sq.cm. per g.[a]			From 3 days to 5½ months	From 3 days to 19 months	From 3 days to 32 months	From 3 days to 58 months
4 by 4 by 40-in. Bars 0 to 3-in. Aggregate							
Modified portland 3CS—51% 3CA—6%	1800	0.8	0.77	+45	+30	..	−20
		1.0	0.65	+30	+25	..	−20
		Avg.	..	+40	+30	..	−20
	2100	0.8	0.77	+5	−15	..	−80
		1.0	0.62	−5	−15	..	−50
		Avg.	..	0	−15	..	−65
	Avg.	0.8	..	+25	+10	..	−50
		1.0	..	+10	+5	..	−35
		Avg.	..	+20	+10	..	−40
Low-heat portland 3CS—26% 3CA—6%	1800	0.8	0.76	0	−10	..	−50
		1.0	0.64	0	−5	..	−50
		Avg.	..	0	−10	..	−50
	2150	0.8	0.78	−10	−20	..	−55
		1.0	0.61	−25	−25	..	−85
		Avg.	..	−20	−25	..	−70
	Avg.	0.8	..	−5	−15	..	−55
		1.0	..	−15	−15	..	−70
		Avg.	..	−10	−15	..	−60
Portland-pozzuolana (25% shale) portland cement composition 3CS - 52% 3CA—6%	2000	0.8	0.86	+25	−30	..	−100
		1.0	0.68	−35	−55	..	−100
		Avg.	..	−5	−40	..	−100
	2250	0.8	0.83	−35	−70	..	−130
		1.0	0.69	−65	−85	..	−115
		Avg.	..	−50	−80	..	−120
	Avg.	0.8	..	−5	−50	..	−115
		1.0	..	−50	−70	..	−110
		Avg.	..	−25	−60	..	−110
6 by 12-in. Cylinders 0 to 1½-in. Aggregate							
Modified portland	1800	0.8	0.85	+5	+5	+10	..
		1.0	0.69	−5	0	0	..
		Avg.	..	0	0	+5	..
Low-heat portland	1800	1.0	0.66	−5	0	0	..
Portland-pozzuolana	2000	0.8	0.94	−5	−55	−75	..
		1.0	0.74	−10	−85	−115	..
		Avg.	..	−5	−70	−95	..

[a] Klein basis.
[b] + indicates expansion.

NOTES:

4 by 4 by 40-in. Bars.—Concrete sealed in thin copper containers immediately after casting. Sponge rubber at ends and asphalt coating inside containers to prevent restraint. Steel plugs in ends of bars extended through ends of containers, with mastic seal. Mass curing from initial temperature of 40 F. to age 5½ months, when several rapid temperature cycles, 80 to 40 F., imposed; storage at room temperature thereafter. Cement composition and fineness, as indicated. Aggregate, 0 to 3-in. gravel, fineness modulus 6.10. Cement contents, as indicated. Slump 2 in., water-cement ratio as indicated. Values are average for 3 specimens.

6 by 12-in. Cylinders.—Concrete sealed in thin tinned-cans immediately after casting. Longitudinal crimps in cans loosened after concrete hardened to prevent restraint of containers. Measurements by fulcrum-plate strain gage on three 10-in. gage lines 120 deg. apart around a cylinder. Gage plugs in concrete extended through holes in cans, with mastic seal. Mass curing from initial temperature of 40 F. to age 6 months; storage at room temperature thereafter. Cement composition and fineness, as indicated in table for bars. Aggregate, 0 to 1½-in. gravel, fineness modulus 5.46. Cement contents, as indicated. Slump ¾ in., water-cement ratio, as indicated. Values are average for 3 specimens.

Although data are lacking to prove the point, it seems likely that in a concrete mass of ordinary composition, the autogenous movement during the first few days would always be of the nature of a slight expansion, since free water would be available in the pores. If a concrete was of such composition that considerable bleeding would occur, sealed concrete specimens from which the bled water had not been removed would be expected to expand for a longer period than similar concrete in a large mass.

The results of observations on sealed concrete bars containing three types of cement, started at the age of 3 days and extended over nearly 5 yr., are shown in Table I and Fig. 2. The table shows values for each of two cement contents and two finenesses of each cement. In the diagram, average values are shown for each type of cement. The movements were measured in a direction *parallel* to the upper surface of the concrete, as cast. During the first few months the concretes were carried through temperature cycles similar to those that would occur within the mass of a large dam made of concrete containing the particular cements. At the age of 5½ months, the specimens were subjected to several relatively rapid changes in temperature for the purpose of determining the coefficient of thermal expansion. Subsequently they were stored at room temperature. The direct effect of temperature has been taken into account in all results shown.

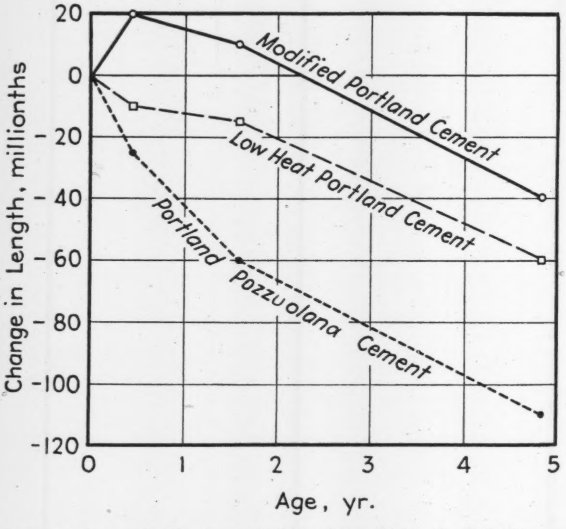

FIG. 2.—Average Length Changes of 4 by 4 by 40-in. Sealed Concrete Bars.

NOTES:
For test details, see notes to Table I—Bars. Values in diagrams are averages for all conditions for each cement.

It will be noted that the bars containing the modified portland cement showed some expansion during the first few months and those containing the portland-pozzuolana cement showed considerable contraction from the beginning. The results are very similar to those shown by Savage[9] up to the age of 90 days for concretes containing portland and portland-pumicite cements. After about 5 months the modified portland cement concrete began to contract, and after a little more than 2 yr. it showed a net contraction. At the end of 58 months, the average net contractions for the modified portland, the low-heat portland, and the portland-pozzuolana cements were about 40, 60, and 110 millionths, respectively. The low-heat cement showed movements intermediate between those of the modified portland and the portland-pozzuolana cements at all ages.

As shown by the data in Table I, there was a tendency for greater contraction with the higher cement content and the finer cements.

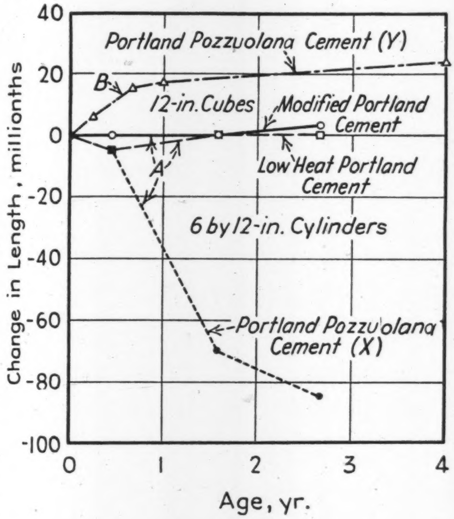

FIG. 3.—Average Length Changes of Sealed Concrete Specimens.

NOTES:

Curves A.—For test details, see notes to Table I—Cylinders.

Curve B.—For test details, see notes to Fig. 1, except as follows: Storage at room temperature after age 3 months. After age 3 months strains measured by 10-in. fulcrum-plate strain gage on horizontal lines on two opposite faces of cube at midheight.

Results of measurements on cylinders made of concretes similar to those discussed above, but having aggregate of 1½-in. maximum size and containing only the coarser cements, are shown for the period 3 days to 32 months in Table I and Fig. 3, curves *A*. The movements measured were *perpendicular* to the upper surface of the concrete as cast. During the early ages, the temperature conditions were similar to those that obtained for the tests on the bars, but the cylinders were not subjected to any large and rapid temperature changes for the determination of the coefficient of thermal expansion.

Up to the age of 32 months, when the tests were discontinued, the specimens containing the modified and low-heat portland cements showed little movement, although the specimens containing the portland-pozzuolana cement showed contractions comparable with those exhibited by the bars. The true cause for the difference in behavior of the bars and the cylinders is unknown, but it is not unlikely that the following conditions had some bearing thereon: (1) direction of measurement with respect to the free surface of the concrete as cast, and (2) for the same cement content the concrete with the smaller maximum size of aggregate was of higher water-cement ratio and hence would have contained more free water.

In Fig. 3, curve *B*, are shown the results of measurements on two sealed 12-in. cubes of concrete containing a portland-pozzuolana cement. The concrete was subjected to mass curing up to age 3 months. In these tests a continued expansion was observed, in contrast to the contraction observed for the portland-pozzuolana cement discussed above. The pozzuolana used in the tests on these cubes was a volcanic sand, calcined and ground before being blended with the cement. The blended cement made from it produced mixes which were very similar to mixes made with straight portland cement in texture, water requirement, and bleeding tendency. The pozzuolana used in the tests, the results of which are shown in Fig. 2 and in curves *A* of Fig. 3, was a shale; mixes containing it were very sticky and had practically no bleeding tendency.

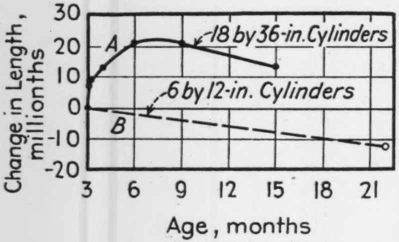

FIG. 4.—Length Changes of Sealed Concrete Specimens.

NOTES:

Curve A.—Specimens, 18 by 36-in. concrete cylinders, moist cured to age 3 months, then sealed in sheet metal containers and stored at 70 F. Measurements by fulcrum-plate strain gage on three 20-in. gage lines 120 deg. apart around a cylinder. Gage plugs in concrete extended through holes in cans; with mastic seal. Cement, normal commercial portland. Aggregate, 0 to 6-in. gravel. Cement content 1.1 bbl. per cu. yd. Slump 1 in., water-cement ratio 0.45 by weight. Values are averages for 2 specimens.

Curve B.—Test details similar to those described in notes to Table I, except as follows: Mass curing from initial temperature of 53 F. to age 3 months; storage at room temperature thereafter. Age at base reading for data shown, 3 months. Cement, modified portland—3CS, 57 per cent; 3CA, 5 per cent. Specific surface, 2100 sq.cm. per g. (Klein basis). Aggregate, 0 to 1½-in. gravel, fineness modulus 5.23. Cement content, 1.1 bbl. per cu. yd. Slump 1 in., water-cement ratio 0.65 by weight. Values are averages for 4 specimens.

The results of some miscellaneous tests on sealed specimens are shown in Fig. 4. Curve *A* shows the movement of two 18 by 38-in. cylinders which were sealed in sheet-metal containers after having been stored for 3 months under moist conditions. The cement was a standard portland. The concrete expanded for several months, probably utilizing what free water was available in the pores, and then began to contract.

Curve *B* shows the contraction from age 3 to 21 months of a group of four 6 by 12-in. cylinders which had previously been used for some thermal expansion experiments. These specimens were sealed immediately after being cast, but measurements for the purpose of determining autogenous movements were not begun until age 3 months. The cement was a modified portland cement of moderately high fineness.

DISCUSSION

Mr. H. S. Meissner.[1]—I should like to elaborate a trifle on the statement that reduction in the volume of water within cement paste occurs as hydration proceeds. In mass curing tests, conducted by the Bureau of Reclamation on large adiabatically-cured concrete specimens hermetically sealed in metal containers, we have observed certain corroborating evidence. These containers had mercury-filled manometer tubes attached to them to register the pressures noted in the concrete or within the containers as the concrete was taken through a rising and, later, falling temperature cycle. The manometer level was adjusted to record zero pressure within the container of fresh concrete at the beginning of the test. The pressure was observed to increase as the concrete set and produced heat and as it (as well as air within the sealed container) expanded with rising temperature. When the concrete was cooled to its initial temperature, the pressure was observed to fall and to indicate a vacuum. This, to my notion, is proof that a reduction in absolute volume of total matter within the container occurred; either the over all dimensions of the concrete were reduced, or the air which occupied the surplus space within the container has in some measure gone into the concrete to occupy newly created voids, thus producing a vacuum in the sealed specimen.

Mr. M. A. Swayze[2] *(by letter)*.—The paper by Mr. Davis is an important contribution since the subject of autogenous volume changes in concrete is one which has received far too little attention in the past in the general study of concrete properties. In the case of large dams, for example, the large autogenous shrinkage shown by Mr. Davis in Fig. 2 for portland-pozzuolana cement will produce just as wide a crack in concrete as would a simultaneous decrease in concrete temperature of about 15 F. Further, the cracking tendency due to autogenous shrinkage will be the greater, since the majority of the shrinkage reported seems to occur after the concrete has developed a high modulus of elasticity, as compared to normal thermal shrinkage which takes place at early ages when the modulus is low and the induced stresses can more easily be accommodated by plastic flow.

Regarding early autogenous volume changes during the setting and early hardening periods, we have all noted the rapid absorption of water at the surface of concrete specimens after the period of sedimentation or "bleeding" has passed. This is a good illustration of the fact that the autogenous volume change at this time is shrinkage, rather than expansion. Since the mechanics of hydration and the character of hydration products are the same as at the late ages, it is, therefore, more logical to conclude that autogenous movement at any age is in the direction of shrinkage. During the water-curing period of normal unsealed specimens the volume of the concrete may actually increase, since we are adding water, and, therefore, volume to the system. However, considering only the closed system, we have autogenous shrinkage rather than expansion at any age. Expansion noted during the first few weeks or months must be attributed to a secondary influence such as expansion in place of grains of free lime, rather than to swelling of the silicate gel, and, therefore, outside the stated scope of Mr. Davis' paper.

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[1] Engineer, Bureau of Reclamation, U. S. Department of the Interior, Denver, Colo.

[2] Director of Research, Lone Star Cement Corp., Hudson, N. Y.

Since the rate of cement hydration and strength development is greatest in the early period, it would, therefore, seem that the time at which initial measurements are taken is of considerable importance. Our research laboratory has given this matter a good deal of attention, both mental and material. Theoretically, the time should be that at which the mass has lost its plasticity, so that horizontal movement may no longer be translated into vertical subsidence. Practically, so many factors must be considered—type of cement, cement content, particle interference of aggregates—that establishment of an exact time for the initial measurement is difficult if not impossible.

In general, however, much earlier ages are indicated by our studies than are generally used. These range from about 6 hr. for cements which hydrate rapidly to 15 hr. or even longer for very slow-hardening cements. Autogenous shrinkage occurring between these ages and the usual time of initial measurement at one, two, or three days may be relatively large, and may, therefore, introduce a serious error in data from which we attempt to deduce the proper type of cement for a special use.

In conclusion, I wish to emphasize the need of knowledge on volume change in concrete at the early ages. Mr. Davis has made an excellent contribution to our understanding of the late changes; the early ones which precede the usual initial readings must also be understood if we are to design concrete structures with certainty. The laboratory which I represent hopes to present a report on this phase of volume change eventually, but a wider consideration is necessary than that which can be given by a single organization.

MEASURING CHANGES IN PHYSICAL PROPERTIES OF CONCRETE BY THE DYNAMIC METHOD

By W. T. Thomson[1]

Synopsis

In investigating the durability of concrete by freezing-and-thawing tests, it has been found that the deterioration of the specimen is accompanied by certain changes in the physical properties of the material. The properties of the material most readily affected by deterioration are the modulus of elasticity and the decrement of vibration, both of which can be measured by the dynamic method. This paper presents tests performed on concrete composed of various aggregates with different water-cement ratios. Included, also, are certain peculiarities of results obtained by the dynamic method.

Two important dynamical properties of any elastic system are the natural frequency of vibration and the damping capacity. In the case of a vibrating beam of given dimensions, the former is related mainly to the ratio of the modulus of elasticity and the mass density, while the latter is related to the energy dissipated by internal and external friction.

During the past few years, the modulus of elasticity of materials such as concrete has been determined by dynamic methods **(1, 3, 10, 17)**[2]. Essentially, this method requires the measurement of the natural frequency of vibration of prismatic bars, from which the modulus of elasticity can be calculated by the various known relations, depending on the nature of the vibrating system employed. Both longitudinal and flexural vibrations have been used, the specimen being vibrated by electromagnetic, electrostatic or by mechanical means.

From the fundamental point of view, the damping capacity of materials such as concrete is of as much importance as the modulus of elasticity, and deserves to be investigated. Excluding external friction, the damping capacity is the amount of work dissipated into heat by internal friction in a unit volume of the material during a cycle of vibration **(12, 13, 14)**. For comparative purposes, the specific damping capacity which is the ratio of the damping capacity to the potential energy or resilience at the same values of the stress is a more convenient term to use. Since the deterioration of material such as concrete is accompanied by an increase of internal friction, it seems quite logical to expect changes in the damping capacity with deterioration.

Probably the most convenient method of determining the specific damping capacity is by measuring the logarithmic decrement of vibration. The logarithmic decrement is defined as the natural logarithm of the ratio of any two successive amplitudes of a vibration whose amplitude decreases exponentially, and can be shown to be equal to one half the specific damping capacity (12, 13, 14). It can also be represented as the resistance divided by the elastic reactance of the system.

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[1] Assistant Professor, Department of Applied Mechanics, Kansas State College, Manhattan, Kans.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1120.

One simple method of measuring the decrement is by observing the sharpness of the resonance curve, a method which was used in this investigation. The logarithmic decrement is quite generally represented by the following approximate relation:

$$\delta = \frac{\pi(f_2 - f_1)}{f_0} \qquad \text{........(1)}$$

where:

δ = logarithmic decrement,

f_0 = resonant frequency of vibration, and

f_1, f_2 = frequencies on either side of resonance at which the amplitude of vibration is $1/\sqrt{2}$ times the maximum.

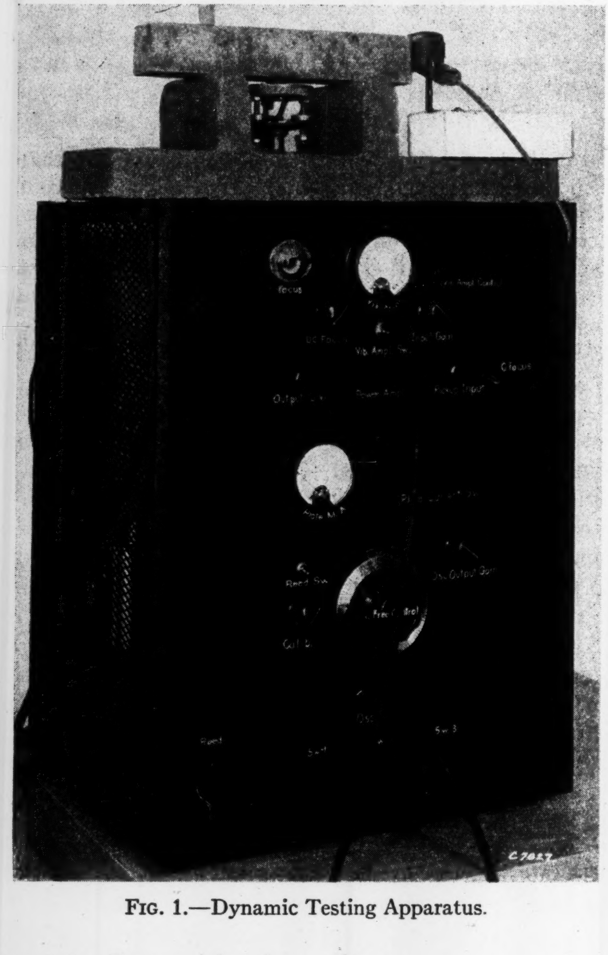

FIG. 1.—Dynamic Testing Apparatus.

Although the decrement varies widely for different materials, the change in decrement or the relative decrement for any given material should be a measure of its deterioration.

APPARATUS

The dynamic testing apparatus used for the investigation consists of a beat-frequency oscillator with a frequency range of 0 to 3000 cycles which is amplified and fed into a magnetic driver, a magnetic pickup connected to an audio frequency amplifier and an output meter which registers the amplitude of vibration. Concrete beams of size approximately 2 by 2 by $11\frac{1}{2}$ in. were supported at its nodal position 0.224 L from its ends, and driven at the center where a thin sheet of galvanized iron $\frac{3}{4}$ in. square cemented on the face acts as an armature. Since the ratio of the amplitude at the ends to that at the center is approximately 1.6 (2), the pickup is placed in contact with the end of the beam. The entire apparatus, including one of the specimens in position, is shown in Fig. 1.

Although any standard oscillator and amplifier may be used for these tests, the following precautions must be observed:

1. Since the magnetic force is proportional to the square of the flux, a direct polarizing flux is necessary in the magnetic driver in order to produce a driving force of the same frequency as the oscillator. For instance, if φ_0 and φ are the direct and alternating fluxes, respectively, the force exerted by the driver is $k(\varphi_0^2 + 2\varphi_0\varphi + \varphi^2)$ and by using a strong polarizing flux, φ^2 can be made negligible compared to $2\varphi_0\varphi$.

2. It is advisable to use a crystal pickup rather than a magnetic pickup, since in many cases the latter generates voltage which is proportional to the velocity rather than the amplitude. If a magnetic pickup of the type mentioned is used, an integrating circuit may be used to convert this voltage in terms of displacement (6).

3. The frequency output characteristics of both the oscillator and amplifier must be constant in order to give the true resonance curve, and the output of the oscillator should be free from harmonics.

TEST PROCEDURE AND EXPERIMENTAL RESULTS

The experimental investigation was performed with an exploratory viewpoint and should be interpreted as representing possibilities rather than final results.

Each group of beams contained one type of aggregate screened to 75 per cent continuous gradation. (Percentage passing each sieve = 75 per cent of preceding sieve.) Water-cement ratios of 7.5, 5.75, and 5 gal. per sack, were used and the mix was varied to give the same slump. After curing for 30 days in the moist room, the beams were subjected to freezing-and-thawing cycles at temperatures of −15 and 80 F. The freezing-and-thawing cycle represents 2 hr. in the freezer and thawing for 30 min. under water at 80 F.

For lateral vibrations of prismatic beams, the following equation or its equivalent may be found in various texts (15, 16):

$$f = \frac{m^2 k}{2\pi L^2}\sqrt{\frac{Eg}{\gamma}}$$

When applied to rectangular beams, the equation for the modulus of elasticity becomes

$$E = \frac{713.9}{m^4}\,\rho\,\frac{L^4}{d^2}f^2 \ldots\ldots (2)$$

where:

E = modulus of elasticity in pounds per square inch,

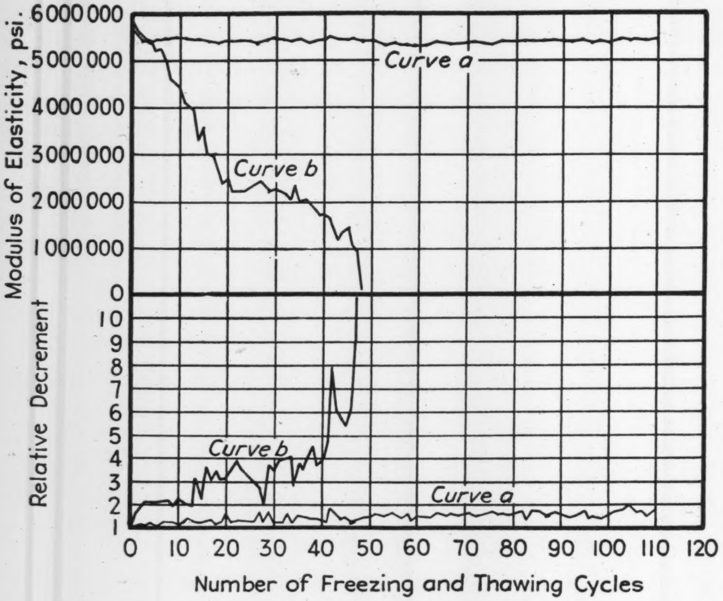

FIG. 2.—Difference in Behavior of Good and Poor Specimen.

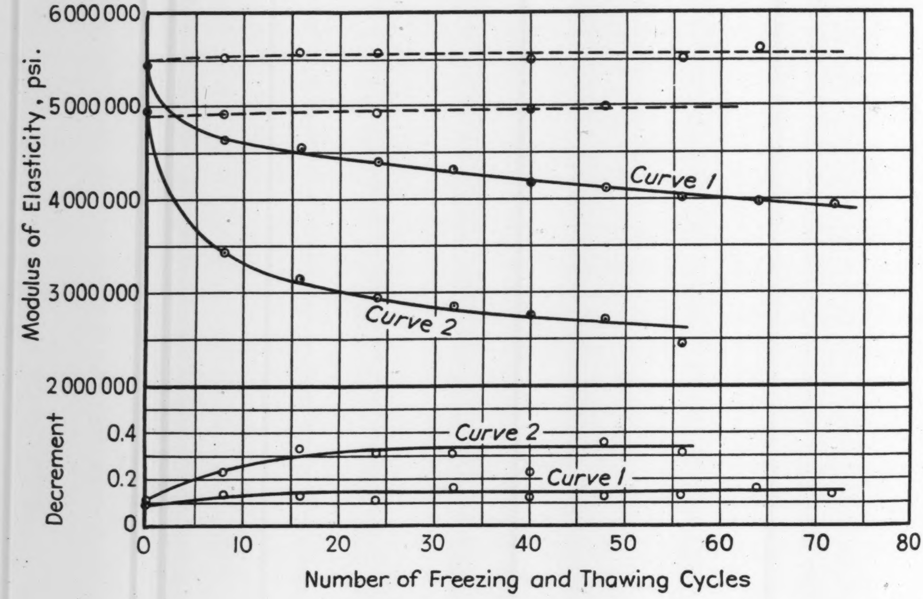

FIG. 3.—Average Deterioration Curves of Concrete with Kaw River Aggregate.
Curve 1, $\frac{w}{c}$ = 5 gal. per sack. Curve 2 $\frac{w}{c}$ = 7.5 gal. per sack.

L = length of beam in inches,

d = depth of beam in inches,

ρ = density of specimen in pounds per cubic foot,

f = resonant frequency in kilocycles,

m = a number depending on $\frac{d}{L}$ (= 4.73 for small ratios of $\frac{d}{L}$), and

$$713.9 = \frac{48\pi^2 \times 10^6}{12^4 \times 32.2}.$$

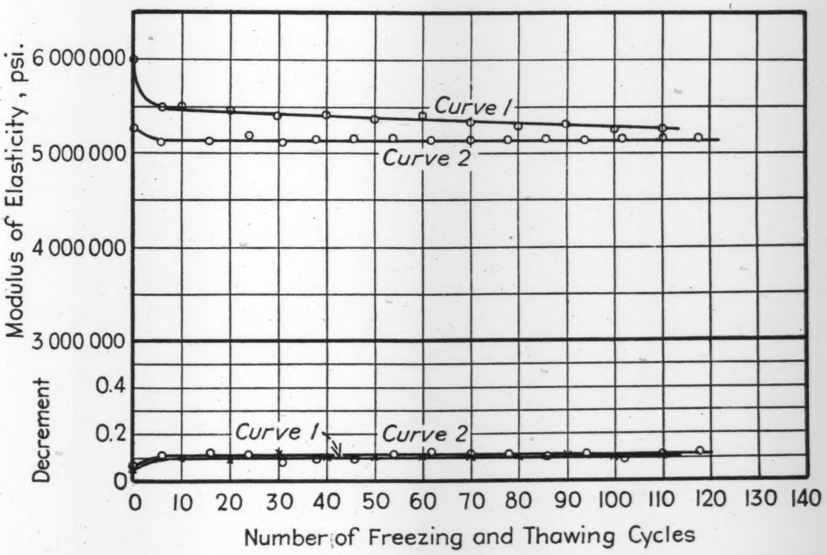

FIG. 4.—Average Deterioration Curves of Concrete with Elgin Aggregate.

Curve 1, $\frac{w}{c}$ = 5.75 gal. per sack. Curve 2, $\frac{w}{c}$ = 7.5 gal. per sack.

The decrement was determined from Eq. 1.

Figure 2 represents typical behavior of a good and a poor specimen, as shown by curves *a* and *b*. In this case the relative decrement, defined as the decrement divided by the original decrement at zero freezing-and-thawing cycles, was plotted. The poor specimen broke in 48 cycles, while the good beam showed practically no signs of deterioration at 110 cycles. Both beams were made of identical material; however, specimen *b* contained one piece of unsound aggregate in the middle.

Figure 3 represents averages of eight Kaw River aggregate (mainly feldspar and quartz) beams with water-cement ratios of 5 and 7.5 gal. per sack. These were compared with control beams (shown with dotted lines) which were continuously kept in the moist room. It is quite apparent from these curves that this aggregate produces poor concrete.

A more satisfactory type of concrete is given by the Elgin aggregate (glacial gravel) beams, shown in Fig. 4. The decrease in *E*, and the increase in decrement is quite small for both water-cement ratios.

All tests performed so far lead to the following conclusions:

1. The largest change in the modulus

of elasticity and the decrement of vibration takes place during the first few cycles of freezing and thawing, and even good concrete will show some decrease in the modulus of elasticity during the period.

2. Between good and poor specimens there is no apparent difference in E or δ at zero cycles of freezing and thawing.

TABLE I.—AVERAGE PHYSICAL PROPERTIES OF MATERIALS BY THE DYNAMIC METHOD.

Material	Water-Cement Ratio, $\frac{w}{c}$ gal. per sack	Aggregate	Mix by Volume	Values at 30 days and zero cycles		Values at 20 cycles		Remarks
				E_0	δ_0	E/E_0	δ/δ_0	
Concrete	5.0	Kaw River	1:3.0	5 460 000	0.0957	0.815	1.5	
Concrete	7.5	Kaw River	1:5.3	4 950 000	0.114	0.612	2.8	
Concrete	5.75	Elgin	1:4.0	6 000 000	0.0610	0.908	1.6	
Concrete	7.5	Elgin	1:5.4	5 270 000	0.0759	0.976	1.5	
Concrete	5.0	Ground Kaw River	1:1.56	3 640 000	0.0756	0.99	1.2	no strength, broke soon after 30 cycles
Concrete	7.5	Ground Kaw River	1:2.0	2 970 000	0.0893	0.98	1.02	
Limestone, fossiliferous				4 800 000	0.032			
Ohio limestone				8 000 000	0.040			
Limestone					0.021[a]			
Steel				29 400 000 to 30 200 000	too small to measure			

[a] Value given in reference (7).

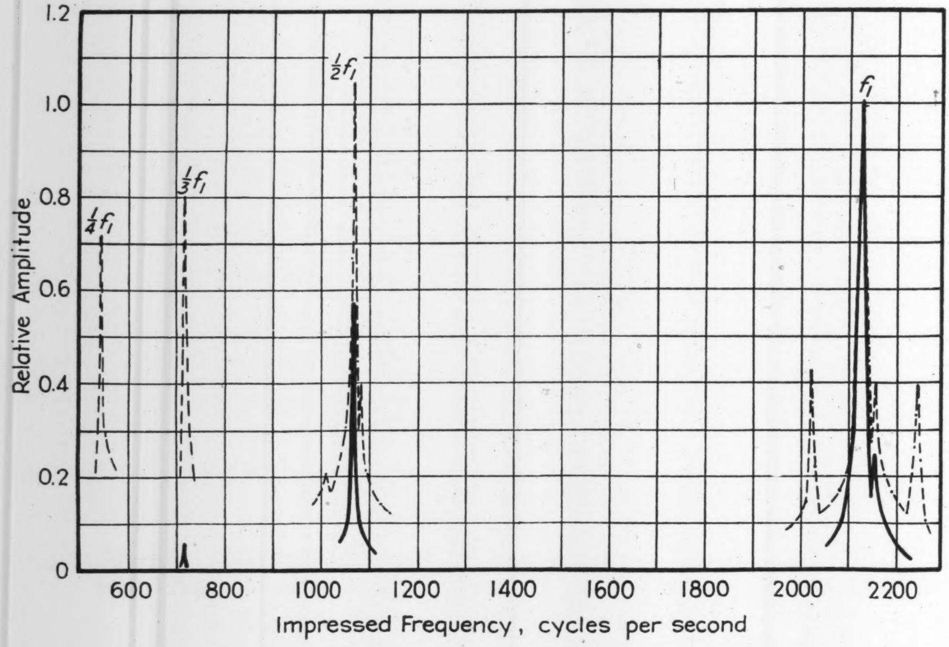

FIG. 5.—Effect of Direct Polarizing Flux on Amplitude Response.

3. Poor specimens can generally be detected within 20 cycles of freezing and thawing, and the changes in E and δ for

such specimens are large as compared with good specimens.

4. It should be pointed out that decrement measurements obtained from the resonance curve is subject to a certain amount of variation (see Fig. 2). One reason for this variation is probably due to the fact that a variation in the internal friction is produced by any rearrangement of the concrete particles in the minute cracks developed by the freezing and thawing. There are also some inaccuracies introduced in reading the width of the resonance curve. Since the smallest value of $(f_2 - f_1)$ is in the neighborhood of 60 cycles, and one division on the Vernier dial of the testing apparatus represented 6 cycles, it is possible to have a variation of 20 per cent in the decrement reading. A one-plate condenser connected parallel to the main tuning condenser would greatly reduce this inaccuracy.

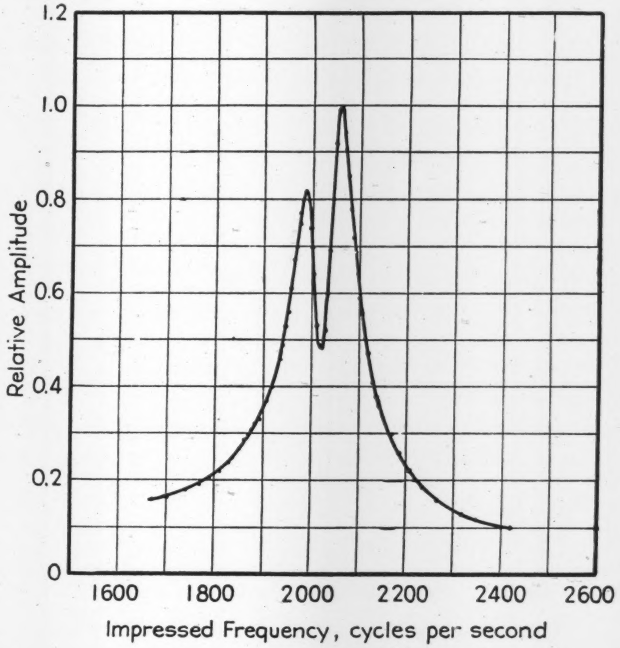

FIG. 6.—Double Hump Characteristics of Certain Concrete Specimens.

Table I gives the results for various materials by the dynamic method of testing materials.

This paper would not be complete without indicating some of the peculiarities observed in the dynamic method of testing materials. These illustrations should also stress the importance of the precautions listed under the section on apparatus.

Figure 5 shows the response curves obtained for a limestone beam with and without the polarizing flux in the magnetic driver. These are indicated by the solid and dotted curves, respectively. Without the d-c. flux, a large response in the amplitude of vibration is indicated at half the resonant frequency of the beam, since the driver attracts the armature during the negative as well as positive parts of the cycle. Minor peaks of response are present on both sides of the resonant frequency, and amplitude peaks at one third and one fourth the natural frequency also occur. When the *d-c.* flux is applied, these minor peaks are eliminated and the responses at $\frac{1}{2} f_1$ and $\frac{1}{3} f_1$ are considerably reduced.

Figure 6 represents the type of resonance curves obtained in some concrete beams which were subjected to a number of freezing-and-thawing cycles. Varying the air gap between the driver and the armature produced no change in the shape of the resonant curves. This gives reason to believe that the peculiarities in the shape of the resonant curves are in the beams themselves rather than in the magnetic driver or the oscillator. Such double curves were found in practically all beams where a crack had become noticeable, indicating a possible origin for the effect. When such beams were encountered it was impossible to calculate the decrement or the modulus of elasticity.

One other item which should be mentioned refers to the sonic method of determining the natural frequency of vibration. Previous investigators have used a method of tapping the specimen with a hammer and obtaining the natural frequency by means of calibrated wires. Although this method is satisfactory on relatively new beams, it should not be used on old specimens. It was found that on striking old beams the natural frequency of vibration was reduced considerably. Tests performed on old beams whose moduli of elasticity have values of about 80 per cent of their initial values, indicated that the natural frequency of vibration may be lowered as much as 10 per cent by being struck with a series of sharp blows.

When testing material such as steel by the dynamic method, one is often confused by the large number of resonant points of vibration. These peaks of response can easily be accounted for by a knowledge of the modes of vibration of prismatic bars. The second mode of vibration will appear at approximately $\left(\frac{7.85}{4.73}\right)^2 f_1$, the third, at $\left(\frac{10.99}{4.73}\right)^2 f_1$, etc. (2). Possibilities of submultiples of these frequencies, as shown in Fig. 5, may greatly add to the confusion.

In conclusion, the author hopes that this paper will stimulate further interest in the dynamic method of testing materials, and that the physical properties of concrete which have been mentioned, will be more thoroughly investigated in connection with its durability.

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DISCUSSION

Mr. M. O. Withey.[1]—As I understand it, Mr. Thomson determines the initial modulus of elasticity in this test?

Mr. W. T. Thomson.[2]—Due to the small amplitude of vibration and the small stresses produced by the dynamic method, the modulus of elasticity calculated from these experiments should correspond to the initial modulus of elasticity. On the other hand, since the effect of internal friction is not included in these calculations, the modulus of elasticity as calculated from Eq. 2 would not represent the true modulus of elasticity where large damping is involved.

Mr. Withey.—What would be the cost of this apparatus in its simplest form?

Mr. Thomson.—The material cost of the apparatus is in the neighborhood of eighty dollars.

Mr. F. E. Richart.[3]—Mention was made of the impact produced when the beam was struck with a hammer to induce free vibration. I would suggest that this impact could be obviated by applying a dead load hung by a string. The load could then be released suddenly, by cutting the string, to produce free vibration of the test beam.

Mr. Thomson.—Mr. Richart's suggestion would do away with impacts and eliminate variation in the modulus of elasticity due to rearrangement of internal particles. However, due to the rapid damping of concrete, one may encounter difficulties in determining the natural frequency of concrete beams by this method.

The Chairman (*Mr. Stanton Walker*[4]).—I wish to inquire of Mr. Thomson whether he has any information on the effect of the moisture content of the test specimen on the results of his test.

Mr. Thomson.—Moisture content of the test specimens would alter the damping factor and apparent density of the beam, thereby altering the calculated values of the modulus of elasticity. I have made no investigations regarding moisture content and am unable to make any statement as to its effect.

Mr. Walker.—Were your specimens saturated?

Mr. Thomson.—The test specimens used in this experiment were saturated and allowed to dry only during the short period when readings were being taken.

Mr. Gerald Pickett.[5]—Mr. Thomson has made a significant contribution by investigating the damping factor as well as the change in frequency with deterioration. I hope this work will continue until the whole resonance curve for forced vibrations is investigated. The shape of the recorded curve depends upon the input, the dimensions of specimen, the physical nature or condition of the specimen and the pickup mechanism. If a specimen changes due to deterioration or other causes, the resonance curve will change. For example, in a qualitative sense general deterioration is accompanied by broadening of the curve and a shift of the peak to the left; localized disintegration, such as might result from the failure of one piece of aggregate or the formation of a crack should be manifested by something like the double hump shown in Fig. 6 of the paper. Such experimental curves are not at present easy to interpret; however, it should be possible to calculate resonance curves representing any assumed physical condition of a specimen, that is, assumptions with respect to the values of Young's modulus of elasticity, internal friction, and localized points of disintegration or weakness. If a sufficient number of such theoretical resonance curves were calculated and made available to investigators, it should be possible to match an actual curve with a theoretical one, and from the assumption made in the calculations determine the physical condition of the specimen.

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[1] Professor of Mechanics, University of Wisconsin, Madison, Wis.

[2] Assistant Professor, Department of Applied Mechanics, Kansas State College, Manhattan, Kans.

[3] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[4] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

[5] Research Physicist, Portland Cement Assn., Chicago, Ill.

If this suggested investigation of the whole resonance curve is undertaken, a word of caution is in order: first, a purely mathematical calculation of theoretical resonance curves is very difficult; second, before actual comparison can be made there must be assurance that the actual input is essentially the same as that assumed in the theoretical calculation.

MESSRS. T. F. WILLIS[6] and M. E. DE REUS[6] (*by letter*).—Mr. Thomson's paper is a valuable contribution in that it again calls to the attention of concrete testing engineers some of the possibilities of the dynamic method of testing, and describes an apparatus for making this test and some of the problems encountered in using it.

About 18 months ago, the Research Division of the Bureau of Materials of the Missouri State Highway Dept. started experimenting with this method. Primarily it was to be used for evaluating the effects of various weathering cycles on concrete specimens. Soon we discovered that education and experience had exposed most of us to only a bare smattering of the fundamentals involved; also that our knowledge of the sources and characteristics of the equipment needed was limited. Particularly was this true of the electrical apparatus required. In the hope that assistance may be rendered to others who find themselves in the same situation, we have outlined below some of our experiences and have described in some detail an apparatus which we have found to be satisfactory.

TABLE I.—EXAMPLE OF REDUCTION OF MODULUS OF ELASTICITY CAUSED BY STRIKING.

Beam	Modulus of Elasticity, psi.[a]		
	Before Striking	After Striking	
No. 1	5 400 000	5 300 000	Never frozen
No. 2	4 400 000	4 100 000	Subjected to various numbers of cycles of freezing and thawing
No. 3	3 200 000	2 600 000	
No. 4	2 600 000	1 900 000	
No. 5	2 800 000	1 800 000	

[a] Determined by the dynamic method with electrical apparatus.

In our first tests, the resonant frequencies of the concrete specimens were determined by comparison with a sonometer such as that described by Powers[7]. However, some difficulty was found in obtaining operators who could tune the concrete specimens to the sonometer, particularly when the elastic modulus of the specimens had fallen off considerably due to continued freezing and thawing. Furthermore, as Mr. Thomson points out, the striking of the specimen with a hammer has in itself an effect on the elastic modulus of the test specimen. For instance, the modulus of elasticity of some laboratory demonstration beams was changed as shown in the accompanying Table I.

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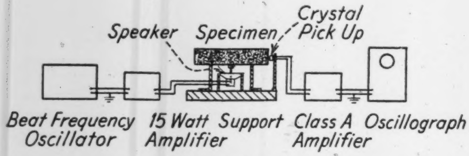
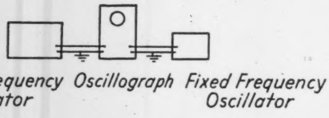
[6] Research Engineer, and Associate Engineer, respectively, Bureau of Materials, Missouri State Highway Dept., Jefferson City, Mo.

[7] T. C. Powers, "Measuring Young's Modulus of Elasticity by Means of Sonic Vibrations," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 460 (1938).

For these reasons it was decided to use the electric method for determination of the natural frequencies of the specimens. After considerable correspondence with the manufacturers of the various equipment items needed and consultation with radio engineers, an apparatus was assembled which has proven to be eminently satisfactory.[8] This apparatus is essentially that described by Hornibrook[9] and differs only in the substitution of a beat-frequency oscillator for the tuned-circuit oscillator and elimination of the decade condenser.



(*a*) For determination of the natural frequency.



(*b*) For calibration of the beat frequency oscillator.

FIG. 1.—Wiring Diagrams.

A wiring diagram showing the hook-up of the various pieces of apparatus is shown in the accompanying Fig. 1. To eliminate electrostatic interference, shielded cable was used for connecting the various units. Two-conductor cable is used to connect the power amplifier to the speaker, but only one-conductor cable is necessary for the other connections as the shielding may be utilized for making the grounded connections.

The fixed-frequency oscillator is used only for calibration of the apparatus and the total cost can be reduced by eliminating this instrument and using another method of calibration which will be described later. Still further reduction in cost can be effected by using a vacuum tube output meter instead of the oscillograph for indicating when the oscillator is vibrating at the resonant frequency of the test specimen. However, substitution of the output meter for the oscillograph would necessitate relying solely on the calibration device built into the beat-frequency oscillator.

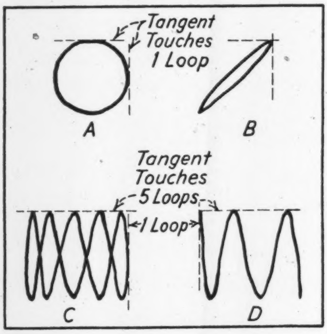


FIG. 2.—Lissajous Figures.

A and *B* are different in phase but both patterns show a 1-1 ratio between the frequencies. *C* and *D* are also different in phase but both patterns show a 5-1 ratio between the frequencies. The pattern shown in *D* has its peaks coinciding.

The beat-frequency oscillator purchased is ordinarily manufactured to cover a range of 25 to 15,000 cycles per second. However, at a small extra cost the manufacturer will modify the instru-

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[8] The following is a list of the various units incorporated in the apparatus, together with the costs. Information concerning the manufacturers of the units may be secured from the Society's headquarters.

	Cost
Beat-frequency Audio Oscillator, Model 79-D Modified	$79.00
15-watt power amplifier, Model 416T	20.00
6-watt Jensen permanent magnet loud speaker, Model PM6C	5.00
Replacement Crystal for Astatic "Tru-tan" phonograph pickup Model B-10	4.00
Class A Amplifier, one stage	15.00
3-in. Oscillograph, Type 164	55.00
Fixed-Frequency (400 cycles per second) Audio Oscillator, Type 813B	36.00
25-ft. one-conductor shielded X'stal microphone cable, Type 8431 10-ft. two-conductor shielded X'stal microphone cable, Type 8402	1.00
Home made support for loud speaker and test specimens	10.00
Total Cost	$225.00

[9] Floyd B. Hornibrook, "Application of Sonic Method to Freezing and Thawing Studies of Concrete," *ASTM Bulletin*, No. 101, December, 1939, p. 5.

ment so that it covers only a portion of this range. Depending on the size of beams which are to be tested, most of the high portion of the range is not necessary and by modifying the instrument the scale graduations are increased in size, enabling more accurate reading of the instrument. For our own instrument, this was modified to cover only the range from 25 to 4000 cycles per second, which makes it possible to read the scale sufficiently accurately so that the probable error is not greater than one-half of one per cent.

Since the frequency stability of a low-cost beat-frequency oscillator is not as good as that of a tuned-circuit oscillator, it is essential that an accurate and quick method for calibration of the oscillator be provided. In our setup the fixed-frequency oscillator in conjunction with the oscillograph serves this purpose. The beat-frequency oscillator is calibrated against the fixed-frequency oscillator by feeding the output voltages of the two oscillators into opposite sides of the oscillograph and observing the resulting Lissajous figures on the screen of the oscillograph. When the frequencies of the two oscillators are the same, the figure will be a circle or an ellipse (see accompanying Fig. 2) depending on the difference in phase. If the frequency of the beat-frequency oscillator is twice that of the fixed-frequency oscillator, the pattern registered on the screen of the oscillograph will show a ratio of two peaks in one direction, either vertical or horizontal, to one peak in the other direction. The ratio of the peaks shown will correspond with the ratio of the frequencies of the two oscillators. This calibration is easily and quickly made and through its use the frequency of the oscillator can be maintained with a maximum variation of less than five cycles per second by making a slight adjustment of the oscillator every 15 min. or so during use of the instrument. The local alternating-current power supply can also be used as a standard for calibrating the oscillator, in lieu of the fixed-frequency oscillator, if the exact frequency of the power supply at the time of calibration is known. However, it is necessary to remember that the instantaneous value of the frequency of the power supply may not always be the same. The calibration device built into the beat-frequency oscillator can also be used; however, it is merely a zero adjustment device which calibrates the instrument at one point only. This is sufficient provided the instrument is in good adjustment but our experience has shown that even a small force exerted on the indicator at the end of its travel may throw out the over-all calibration of the dial, and for this reason a method of calibration which enables checking of more than one point on the dial is to be preferred.

In the first assembly of our apparatus difficulty was encountered in determining the resonant frequency of some test specimens due to the fact that there were often two peaks registered on the oscillograph, which occurred at different frequencies. After considerable experimenting with various parts of the apparatus it was finally discovered that the curve relating the voltage output of the pickup unit (which was of a type that is used for studying vibrations in industrial machinery) to the frequency had a sharp hump over a narrow frequency band. If the resonant frequency of the specimen being tested was close to or within this band it was often impossible to tell the exact natural frequency of the specimen. From the above it is obvious that the voltage output-frequency curve of the pickup unit should be as flat as possible throughout the frequency range which will be encountered in carrying out tests. In our own case, we use $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16-in.

specimens, tested so that the vibrations are in the plane of the short dimension. The fundamental frequency of a normal beam is in the neighborhood of 2000 cycles per second which, after freezing and thawing for a number of cycles, may fall off to as low as 200 cycles per second. The crystal pickup unit which we used had the hump in the voltage output curve at around 1600 cycles per second. At the suggestion of Mr. Floyd B. Hornibrook, we tried an ordinary phonograph replacement cartridge of the crystal type which has proven to be very satisfactory as well as much cheaper than the original unit.

Another point in the construction of this apparatus which should be carefully considered is the natural vibration period of the stand used in supporting the specimen. This should fall outside the range within which the fundamental frequencies of the test specimens may lie; otherwise it will interfere with the determination of those frequencies near the resonant period of the stand. This can be prevented by damping the vibrations of the stand by the use of sponged rubber supports for the specimen.

Another method of accomplishing the same purpose is to make the stand heavy and stiff enough that its natural frequency is above the range to be used in testing specimens or light and flexible enough that its natural frequency is fairly low. In the first case some slight interference may be encountered if the fundamental frequency of the specimen falls near one-half the natural frequency of the stand since the stand will vibrate when excited at one-half its natural frequency. This action is similar to the response curves for a beam shown by Mr. Thomson in his Fig. 5. In the second case, there is a possibility that a harmonic frequency of the stand may cause some slight interference. If the interference is not severe it is not necessary to damp it out since the operator soon becomes familiar with the interference points and can make the proper interpretation of his results.

In our own case, the stand shown in Fig. 1 is made up of a base consisting of an 8-lb. 6-in. channel iron 20 in. long to which are bolted the support for the speaker and two 4 by 6 by 6-in. angle irons. The natural frequency of this support in a vertical plane is 470 cycles per second. There is no interference from any harmonics but there is interference at the natural period which, however, is low enough that when a beam has deteriorated to the point that its natural frequency is near this value it has lost about 90 per cent of the original elastic modulus and ordinarily we are not interested in tracing further deterioration.

Mr. Thomson refers to another point which he says may be misleading, that is, the "double hump" characteristic referred to in his Fig. 6 which he attributes to cracking of the beam specimen. We have tested numerous beams which, as a result of freezing and thawing, have shown a noticeable amount of cracking, but it has been the exception for any of these beams to exhibit the characteristic Mr. Thomson describes. In the few cases where specimens have exhibited this characteristic there has been a loss from the original elastic modulus of more than 80 per cent.

We have accumulated a considerable mass of data relating to the disintegration of concrete as caused by freezing and thawing and measured by the dynamic test method. While there remain a number of factors to be investigated, it is already apparent that this test is of considerable value for, as Hornibrook[9] pointed out, there is a relationship between the loss in elastic modulus as determined by the dynamic method and the loss in flexural strength of concrete subjected to repeated freezing and thawing. Our data indicate this relationship to be very definite as is shown in the accompanying Fig. 3. This figure shows the results from a number of series of tests in which were included the following variables: eight different coarse aggregates of similar gradation made into concrete having a cement factor of 1.45 bbl. per cu. yd. and a water-cement ratio of 0.77, three different freezing temperatures, four different thawing temperatures, and five different numbers of cycles of freezing and thawing, namely, 0, 2, 10, 20, and 40 cycles. The percentage loss in modulus of elasticity, plotted as the abscissa, is that calculated for a test on an individual specimen referred to the test on the same specimen just prior to the start of freezing and thawing. The loss of flexural strength, plotted as the ordinate, is calculated by comparing the modulus of rupture of an individual beam to the average of four control beams which were tested before freezing and thawing started. This explains why some of the points show a minus loss in flexural strength. The points grouped about the zero abscissa show the variations in flexural strength of the control beams.

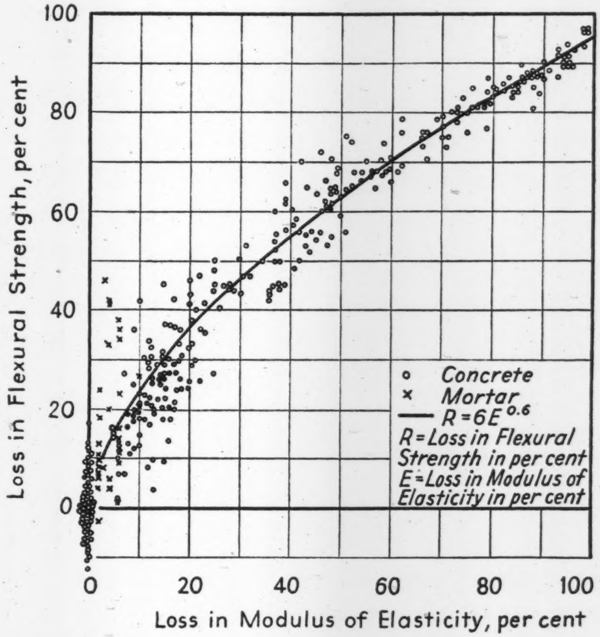

FIG. 3.—Relationship of Loss in Flexural Strength to Loss in Modulus of Elasticity. Each point represents one specimen.

The fact that all the points fall in a narrow band on either side of the average curve, even though they represent tests made on concrete with different constituents, shows the close relationship between these two characteristics of the concrete.

The points deviating most widely from the average curve are those derived from tests on mortar specimens. These fall above the average curve. Why this is true we are not prepared to state at the present time. A possible explanation is that, in the case of mortar specimens, the freezing-and-thawing action merely causes a deterioration of the bond between the cement matrix and the sand particles, which does not increase the discontinuity within the specimen sufficiently to be reflected in the loss in elastic modulus but does cause a loss of flexural strength. Should this be true, the method opens up possibilities for the study of the effect of various weathering cycles or admixtures on the bond between the aggregate and the cement paste. The fact that the curve does not pass through the point of 100, 100 per cent loss suggests that after a concrete has lost all of its elasticity there still is some mechanical interlocking of the particles.

TABLE II.—TYPICAL VARIATIONS IN THE MODULUS OF ELASTICITY OF COMPANION SPECIMENS OF CONCRETE.[a]

Number of Specimens	Natural Frequency, cycles per second	Modulus of Elasticity, psi.	Deviation from Average, per cent
1	2020	5 670 000	3.1
4	2030	5 730 000	2.1
3	2040	5 780 000	1.2
9	2050	5 840 000	0.2
5	2060	5 900 000	0.9
4	2070	5 950 000	1.7
1	2080	6 010 000	2.7
1	2090	6 070 000	3.8
Total 28	Average	5 850 000	1.25

[a] Concrete was 28 days old, had been curing in water, and had never been frozen.

The test is sufficiently sensitive that the small disparities between apparently similar beams can be measured. An example of this is shown in the accompanying Table II in which are shown the results of tests on 28 companion beams. At first it seemed that the deviations shown were due to inherent errors in the test method. However, on rechecking these beams the following day, identical results were obtained for each beam which definitely indicates that the variations were due to some difference in the inherent qualities of the individual specimens and not to errors in the test itself.

As a further indication of the value of the test in showing variations in companion specimens, the accompanying Fig. 4 is shown. In this figure the modulus of elasticity is plotted against the number of cycles of freezing and thawing to which these four specimens were subjected. The unique point indicated by the figure is that, based on the strength results at the end of 40 cycles, specimen No. 4 is stronger than specimen No. 3 and it would be assumed that this had been the case at fewer cycles. The dynamic test, revealed that up to 20 cycles, specimen No. 3 was stronger than specimen No. 4. In general, however, it has been found that companion specimens tend to maintain, throughout a series of freezing-and-thawing cycles, the order which they assume after a very few cycles. In addition, the curves are of value as being typical of the

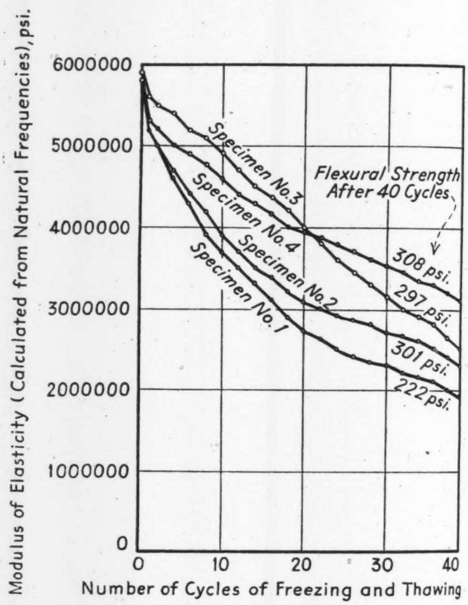

FIG. 4.—Illustrating Observed Variations of Four Companion Specimens.

deterioration, under continued freezing and thawing, of concrete containing a coarse aggregate that, according to service records, is relatively sound, also in showing the variations which may be encountered in test results on companion specimens.

It appears then that, when concrete is frozen and thawed, the loss in flexural strength can well be estimated by the dynamic method of determining the loss in modulus of elasticity; in addition, the dynamic method, of itself, is a means of evaluating the variable reactions of concrete to freezing and thawing. As with other methods of testing the quality of concrete, results should be based on three or more similar specimens so that the extraneous variables of fabrication, curing, etc., may be averaged out.

MR. THOMSON (*Author's closure*).—In writing this paper, the author had in mind a general description of the dynamic method of testing and some of its anticipated possibilities. Mr. Pickett has pointed out another possible approach to the investigation of the resonance curve. It is also gratifying to note that Messrs. Willis and DeReus have accumulated much information in this field.

A few points of interest need clarification: The first refers to the double hump resonance curves found in some beams. The stand used for the support of the beam and driver in the apparatus described was molded from a rubber composition, and its natural frequency of vibration is in the order of a few cycles per second thereby excluding the possibility of its interference with the response of the beam. The double hump characteristic shown in Fig. 6 was independent of the type of pickup used and is inherent in the beam itself. This was also verified by testing beams with natural frequencies in different regions of the frequency scale. As pointed out by Messrs. Willis and DeReus, the cracking of the beams may not be the cause of the double hump. Further experiments have shown that some new beams possessed this characteristic. Possibly a nonuniform distribution of the aggregates or voids may account for this condition.

The matter of the pick-up is of great importance if a true amplitude curve is desired. For the measurement of the natural frequency only, it is of no consequence, since an indication of resonance will be sufficient whether it be amplitude, velocity or acceleration. It should be pointed out that if a crystal cartridge is fastened with a rubber directly to the end of a vibrating beam, the response is that of acceleration and not of amplitude.

The author is in agreement with Messrs. Willis and DeReus in regard to mortar specimen. Ground Kaw River beams, shown in Table I, show that there is very little change in modulus of elasticity and decrement, although the beam is in a deteriorated condition with low flexural strength.

Considering the fact that we are treating a nonhomogeneous material such as concrete by idealized equations based on homogeneity and no friction, it is apparent that extreme accuracy in the measuring apparatus is not justified. Frequency stability of the oscillator and a linear response of the amplifier proportional to the amplitude is much more important than the exact reading of the frequency, since the important items are the change in the natural frequency and the decrement of vibration. Decrement investigations are being continued, and a new apparatus to produce and record damped oscillations of beams is near completion.

SOME PROPERTIES OF MORTARS IN MASONRY

By F. O. Anderegg[1]

Synopsis

An experimental study has been made of some of the properties of masonry mortars with specimens fabricated in contact with masonry units. The properties include: the effect of wetting and drying on volume change, bond and compressive strengths, and durability. The mixes used ranged from straight portland cement 1:3 mortar to straight lime 1:3 mortar, and the consistencies have in general been adjusted to give three flows—70, 105 and 135 per cent. Several different limes have been included, sometimes as well-soaked putties and sometimes as dry hydrates. The results obtained have been compared with similar results with specimens fabricated in metal molds. The effect of the combined packing and suction in altering the properties of the mortars, especially of those higher in lime is shown.

A procedure has been developed for accurate estimation of the extent of bond, using a dye solution. With the aid of this procedure a method for fabricating two-brick beams has been devised which has yielded 28-day results of good agreement. This method is suggested as the basis for acceptance tests for masonry mortars.

A method is described for cutting square, flat specimens from brick joints for determining crushing strengths. By applying suitable shape correction factors, the data may be compared with the results from cubes. Four methods have been used for durability determinations, namely, artificial freezing and thawing, natural freezing and artificial thawing, exposure in a pan on the roof, and a modified sodium sulfate test.

Since volume changes in mortars caused by moisture variations are much greater in specimens prepared in metal molds than in actual masonry, and since the strength contributed by mortars to masonry strength is not proportional to the compressive strength of cubes molded in metal, the whole philosophy of testing masonry mortars needs careful scrutiny. The present investigation was undertaken to develop laboratory tests that will more accurately predict and evaluate the behavior of mortar in masonry, and to add to our present knowledge of the factors governing volume change, bond and crushing strength of mortars and their durabilities, and of the effect of different kinds of lime in varying proportions.

Preliminary studies yielded results of great interest. These included observations that the strengths of cubes made in metal molds from different mixes with a given cement, lime, and sand, varied two to three times as much as the crushing strengths of mortars taken from brick joints. It was also found that the wetness of the mortar is the most important single factor in securing complete contact with an absorbent unit, while the best bond and crushing strengths were obtained with brick of medium absorption in combination with medium consistency mortars. Based on these observations, additional specimens have been prepared and studied for volume change, for bonding and crushing strengths, and for durability.

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[1] Consulting Specialist on Building Materials, Newark, Ohio.

A number of limes were used, and important information concerning their behavior has been obtained. Will a "high-yield" lime with, say, four volumes of water to one volume of solid hydrate in the putty, produce more mortar than a "low-yield" lime? Will the bond strength, the crushing strength, the volume changes on alternate wetting and drying, and the durability be adversely affected due to a higher ratio of water to cementitious material? Can any evidence be secured of possible harmful effect from the continued hydration of MgO in dolomitic lime? These are some of the questions which are demanding attention.

MATERIALS

Brick:

Two types—formed with and without deairing—of stiff mud, side cut, shale brick were selected. The absorption of the first type was such that with the brick flat in ⅛ in. of water the weight absorbed in 1 min. varied from almost none to about 25 g. The absorption of the second type covered the range from 3 to 70 g. Variations in absorption depended upon the burning temperature, and experiments were conducted on brick having 1-min. absorptions in the following ranges: 0 to 1 g., 7 to 13 g., 16 to 24 g., and 50 to 60 g. Typical absorptions after 24-hr. and 5-hr. boiling, for brick having 1-min. absorptions in the above ranges were 0.8 and 1.1, 4.5 and 6.5, 8.9 and 11.7, and 18 and 22.7 per cent, respectively. The saturation coefficients then were about 0.73, 0.69, 0.75, and 0.79, respectively. The wire cutting produced a fairly rough surface, which undoubtedly contributed some mechanical keying to aid the bond, but this factor probably is less important on breaking in cantilever.

Brick *E* of a previous investigation[2] corresponds to the brick of lowest absorption rate used here. Bricks *C* and *D* were equivalent in behavior to a brick having a 1-min. absorption of 20 g., which seems to be about the optimum from the standpoint of the bricklayer. The highest range used here was approximately intermediate between bricks *A* and *B* of the earlier work.

Cement:

A portland cement conforming with A.S.T.M. Standard Specifications for Portland Cement (C 9 – 38)[3] was used.

Lime:

Several different limes, all conforming to A.S.T.M. Standard Specifications for Quicklime for Structural Purposes (C 5 – 26)[4] or for Hydrated Lime for Structural Purposes (C 6 – 31),[5] were selected. Some of their properties were as follows:

Brand	Source	Type	Water in Putty at Normal Consistency at 24-hr., Approx. per cent by Weight	Water for 105 per cent Flow in a 1:2:8 Mix, per cent
A....	Ohio	Dolomitic dry hydrate	45	dry 19.5
B....	Ohio	Dolomitic finer dry hydrate	50	dry 18.2 putty 23.4
C....	Pa.	Dolomitic dry hydrate	45	dry 20.3
D....	Pa.	Dolomitic dry hydrate "Completely hydrated"	40	dry 20.0 putty 23.5
E....	Pa.	High-calcium dry hydrate	55	dry 20.5
F....	Pa.	High-calcium pulverized quicklime	60	putty 31.7
G....	Ohio	Low-magnesian dry hydrate	55	dry 18.8

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[2] F. O. Anderegg, "Watertight Brick Masonry," *Architectural Record*, p. 208 (1931).

[3] 1939 Book of A.S.T.M. Standards, Part II, p. 1.

[4] *Ibid.*, p. 30.

[5] *Ibid.*, p. 32.

The data in the last column were obtained with 1:2:8 mixes containing silica sand, after adjustment at the age of 1 hr. to the given flow. The mortar yields can be calculated from the above figures, lime *F* producing about one-fourth more mortar than the dry hydrated lime *B*, for instance. This "high-yield" putty *F* will carry more sand, so the tendency in the field is to produce a leaner mortar with this lime, which might have some advantages and some disadvantages. Limes *A*, *B*, and *C* showed considerable expansion in the autoclave test,[6] lime *G* a little, while limes *D*, *E*, and *F* were nearly free from expansion.

Sand:

The sand used in fabricating specimens for volume change and durability was a washed, Newark, Ohio, river sand, composed of about one-third glacial sand and about two-thirds sand from local outcroppings of sandstones. The color with the 3 per cent sodium hydroxide test for organic impurities was between fair and good. The percentages passing the standard sieves Nos. 200, 100, 50, 30, 16, and 8 were, respectively: 1.1, 2.5, 9.5, 45.0, 86.7, and 98.9. This sand was also used in the preliminary work on bond and compressive strength of mortar taken from the joints. The silica sand from which mortar was mixed for the bond and compressive strengths was composed of 20 parts of run of mine Ottawa sand, 9 parts of standard Ottawa sand, and 6 parts of local silica sand sized between No. 10 and No. 20 sieves. This last size of sand improved the workability greatly, since the Ottawa sands by themselves feel "dead." At the maximum bulking, "loose and moist," a cubic foot of the Newark sand contained 74.3 lb. of dry sand and of the silica sand 76.5 lb.

Proportions.—In making up mixes, one unit volume of sand was taken as weighing 75; a unit of portland cement, 94; and one of dry hydrated lime, 40.

VOLUME CHANGES CAUSED BY WETTING AND DRYING

Mortars were prepared in 1:1:6, 3:5:21, and 1:2:8 mixes with portland cement, different limes, and the local sand. They were mixed mechanically for 10 min., and brought to a consistency of (*X*) of 70 per cent flow as determined on the flow table. Part of this mortar was placed between two pairs of bricks having a 1-min. absorption of from 0 to 1 g., a strip of very thin tissue paper being interposed to facilitate removal of the mortar slab from the brick. A strip of oiled metal $\frac{3}{8}$ in. wide was inserted in the mortar 1 in. from one edge to aid in securing a strip of mortar suitable for making squares for crushing tests. The joints were pounded to a thickness of $\frac{1}{2}$ in. Small brass inserts were placed in the ends of the mortar joints for later measurements of volume change. The surface of the mortar was well tooled with the point of the trowel.

The remainder of the mortar was brought to a flow of from 103 to 108 per cent (*Y*), and two pairs of bricks absorbing approximately 20 g. in 1 min. were similarly treated. The last part of the mortar was then brought to a fluid consistency of 135 per cent flow (*Z*) and used with two pairs of bricks having a 1-min. absorption of from 50 to 60 g.

After storing for seven days between the bricks in the air of the laboratory, the mortar was removed and the length of the slabs measured to 0.0001 in. The slabs were then exposed to free circulation of air at a temperature of approximately 70 F. By the end of six days the slabs were losing very little, if any, weight, and so were measured again and then immersed in water for 24 hr. at room temperature. This cycle was repeated weekly. In Table I averages are presented in thousandths per cent (parts per hundred thousand), for the number of mortar specimens listed at the head of each column. The use of averages conceals no information, since no significant differences were observed to result from varying the proportions of cement and lime between the limits 1:1 and 1:2. A few additional tests (not tabulated) were run on mortar slabs fabricated from portland cement and sand, 1:3, as well as from the same mix with 10 per cent admixture by volume of lime *B* putty. The volume change of these specimens checked those measured on the 1:2:8 mortars within the error of the measurements.

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[6] Proposed Method of Test for Autoclave Expansion of Portland Cement, *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 297 (1938).

TABLE I.—CHANGES IN LENGTH OF 8-IN. MASONRY MORTAR SLABS CAUSED BY WETTING AND DRYING.

Cycles of 24-hr. soaking and 6-days drying were used.

(Values in thousandths per cent.)

	Lime *A*	Lime *B*	Lime *C*	Lime *D*	Lime *E*	Lime *F*	Lime *G*	Metal Molds	Left in Brick
Number of specimens	12	42	18	24	18	18	18	16	16
First shrinkage	90	80	81	80	78	82	81	163	21
First expansion	55	45	50	40	53	57	50	74	19
Second shrinkage	60	62	61	59	70	74	61	89	20
Shrinkage and expansion at 2 months	44	43	47	45	50	51	47	58	20
Shrinkage and expansion at 4 months	37	36	38	35	41	42	40	56	23
Shrinkage and expansion at 6 months	43	45	50	40	44	47	55	65	19
Net change, 6 months, wet to wet	+10	−3	+15	+6	+10	+8	+9	−43	−1

Another series of 16 slabs ranging from straight 1:3 portland cement to 1:2:8 mortars was made with hydrated dolomitic lime *B*, or with high calcium lime *E*. The slabs were made in metal molds and measured in the same way as for the series just preceding, the results being found in the next to the last column of Table I. At the same time, part of the mortar was placed between bricks with no paper interposed, and in the last column of Table I the results are given for changes in length of the mortar slab while constrained between two bricks.

The lengths of the slabs corresponded in general to those of the brick, ranging from a little less than 8 in. to nearly 9 in. Readings were made at a temperature of 70 ± 2 F. and the gage was checked with a standard bar before and after measuring each group of specimens. The use of a micrometer head, while perhaps not so sensitive as a dial, has certain advantages in giving results constant to within about 0.0003 in. In this way the sticking often encountered with dials was eliminated. A dial reading to 0.0001 in. was discarded in favor of the micrometer head. A standard bar was used frequently to check the position of the micrometer.

Conclusions:

From observing the changes in length during the first six months, caused by wetting and drying 132 mortar slabs fabricated between bricks and removed therefrom at 7 days, it can be concluded that, for a given mix, the changes in length for the three combinations, *X*, *Y*, and *Z* of the consistency of the mortars and absorption of the brick were constant within the error of measurement. The changes in length for cement-lime ratios ranging from 1:1 to 1:2 were also constant within the error of reading.

The greatest observed shrinkage occurred during the first week after the slabs were removed from the bricks, and the second greatest during the drying period in the second week after removal. Data on volume changes of mortar in brickwork starting immediately after contact with the units are needed and will be obtained as soon as possible.

The expansions on wetting, and shrinkages on drying after the first two cycles were of the order of 40 to 50 thousandths per cent. However, at the end of six months, the length of most of the wet slab was apparently slightly greater than when freshly removed from brick. This observation is in accord with previous experience of the author with several thousand 1:3 portland cement specimens undergoing similar cycles, the earlier shrinkage being compensated for as the cement continues to hydrate and expand.

Mortar fabricated in metal molds shrank more initially, and exhibited greater volume changes than similar mortar taken from between bricks. At the end of 6 months, these slabs averaged about 40 thousandths per cent shorter than when first measured.

The change in length of mortars left in contact with brick (no paper between) was much lower, being of the order of 20 thousandths per cent during the given cycle.

The conclusions of Davis and Troxell,[7] and of the author[2] as to the great importance of measuring volume changes of masonry mortars only in brick assemblages are confirmed. The author observed on a series of wall panels at Mellon Institute an initial shrinkage, between the time the mortar set sufficiently to permit use of a strain gage on the reference points and the time when a practically constant value was reached, of 60 thousandths per cent of the mortar thickness, as the average of a large number of panels built with three kinds of brick having low, medium, and high absorption. The change on spraying for 2 hr. was 17 thousandths per cent of the mortar thickness, essentially the same as that observed after a hard 24-hr. rainstorm. This last value is comparable with the data recorded in Table I for mortar measured lengthwise between bricks and with data obtained during the past 9 months for a series of 8 brick pairs for which the change in the thickness of 16 mortar joints was measured over 10 cycles of wetting and drying as 22 thousandths per cent of the mortar thickness.

These experiments have failed to develop any evidence of the expansion of mortars made from lime containing incompletely hydrated magnesia. To explain this observation, the suggestion is made that sufficient moisture is present, especially with weekly soakings, to hydrate a large part of the magnesia rather early. According to Wells and Taylor,[8] about 85 per cent of the magnesia should be hydrated at room temperature within one month, if kept wet. Moreover, calculation will show that when 1 mole of magnesia reacts with 1 mole of water, a net shrinkage in volume takes place, the $MgO + H_2O$ occupying less volume than the $Mg(OH)_2$ formed. When the mortar sets, there are surely present many moles of water for each one of free MgO. This viewpoint does not prove that expansion cannot take place, but does seem to explain the apparent integrity of practically all masonry walls containing dolomitic lime.

Compressive Strengths of Mortar Used in Volume Change Measurements:

From the 1-in. strips mentioned in the preparation of specimens for the volume change measurements, at least 12 squares were clipped for each test condition, the large number being desirable because of the scatter among the results. This scatter can be reduced by skill in surfacing, but it is suggested that variations of the order of ± 15 or 20 per cent are inherent in the mortar itself. The distribution of the capillaries in the brick is variable, so that the suction is greater in some spots than in others.

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[7] Raymond E. Davis and G. E. Troxell, "Volumetric Changes in Portland Cement Mortars and Concrete," *Proceedings*, Am. Concrete Inst., Vol. 25, p. 210 (1929).

[8] Lansing S. Wells and Kenneth Taylor, "Hydration of Magnesia in Dolomitic Hydrated Limes and Putties," *Journal of Research*, Nat. Bureau Standards, Vol. 19, p. 233 (1937).

TABLE II.—COMPRESSIVE STRENGTH OF MORTARS FOR VOLUME CHANGE STUDY.

(Corrected to Cube Shape; Values in pounds per square inch.)

Combination *X* was mortar of 70 per cent flow on brick with less than 1-g. absorption in 1 min. Combination *Y* was brick with 20-g. absorption in 1 min. and 105 per cent flow mortar. Combination *Z* was 135 per cent flow mortar on brick with 50 to 60-g. absorption in 1 min.

The ratio of average dry hydrate strength to putty strength = 1.11, practically the same as the reciprocal of the ratio of the mortar volumes.

Lime	Mix	7 Days Between Bricks			28 Days; 7 Between Brick, 21 in Envelopes		
		X	*Y*	*Z*	*X*	*Y*	*Z*
A Dry Hydrate	1:1:6	1210	1620	1310	1320	2200	1910
	1:2:8	740	1040	1020	840	1340	1270
B Dry Hydrate	1:1:6	1070	2010	1600	1880	2550	1580
Putty	1:1:6	1190	2000	1190	1560	2280	2260
Dry Hydrate	3:4:21	1070	1670	1590	1360	1400	1180
Putty	1:2:8	920	1160	1400	1110	1250	885
C Dry Hydrate	1:1:6	740	1300	1250	870	1900	1740
	3:4:21	545	1060	900	670	1600	1400
	1:2:8	450	900	870	500	960	930
D Dry Hydrate	1:1:6	850	1010	980	890	1250	1210
Dry Hydrate	3:4:21	830	1200	1090	780	1500	1120
Putty	3:4:21	630	1220	980	660	1110	1080
Dry Hydrate	1:2:8	870	1090	930	840	1020	960
E Dry Hydrate	1:1:6	930	1690	1450	960	1970	1670
	3:4:21	730	940	860	740	1030	1010
	1:2:8	270	600	680	560	1230	790
F Putty	1:1:6	890	1510	1230	1130	1610	1210
	3:4:21	550	750	680	590	875	850
	1:2:8	430	680	530	480	720	640
G Dry Hydrate	1:1:6	1300	1420	1300	1700	1780	1740
	3:4:21	1250	1320	1190	1170	1460	1910
	1:2:8	750	980	900	810	1100	950
Average		$Y/X = 1.56$ $Z/X = 1.41$			$Y/X = 1.57$ $Z/X = 1.40$		

Part of the squares were surfaced and tested at 7 days, while the remainder were stored in envelopes on the laboratory shelf until 28 days old, when more specimens were prepared and crushed.

The surfacing operation consists in squaring the edges by rubbing in damp sand on a file. The flat sides are then made plane and parallel with a little moist neat cement paste on a high-speed rubbing table. After a little practice, this can be done quite rapidly and accurately. Correction from the flat shape to a cube shape is made with the factors given in the A.S.T.M. Standard Method of Securing Specimens of Hardened Concrete from the Structure (C 42 – 39).[9] These factors were confirmed by crushing 20 cubes and 20 flat specimens made from the same mortar. The dimensions of the square are read to the nearest 0.02 in. (or 0.5 mm.), and at the same time the thickness is noted. This thickness divided by the smaller flat dimension is the slenderness ratio s. The correction factor C is conveniently calculated from the equation:

$$C = 1.00 - (1.00 - s)/1.2$$

or taken from a chart of this equation. The results are given in Table II.

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[9] 1939 Book of A.S.T.M. Standards, Part II, p. 320.

Best results were obtained with medium consistency mortars in combination with a brick of not too high absorption rate, confirming previous observations made with the help of a large number of masons. Poorest results were obtained with the mortar of lowest flow in contact with low-absorption brick. These results are not as high as they would have been had the paper been left out, judging from parallel experiments with squares cut from mortar fabricated with and without paper between the volumes of the mortars. The distension of the mortar by the water required to produce working consistency, in conjunction with water-retaining capacity, probably controls the effect of the lime on the strength of the mortar.

BOND AND COMPRESSIVE STRENGTHS OF MORTAR IN MASONRY

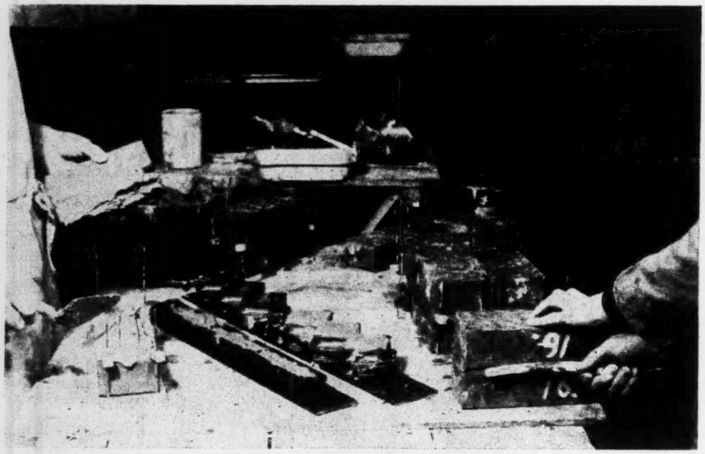

FIG. 1.—Assembly of 2-Brick Mortar Beams to Give Consistent Results.

Contact with the brick is made with a wiping motion. Note metal frame placed in mortar to facilitate later square cutting; also spacers used to control joint thickness. Some of the volume-change slabs may be seen in the upper background.

The bonding strength of a given mortar is of great interest, but it cannot be determined with any degree of accuracy if the contact between the mortar and the brick. It is suggested that the combined actions of compacting and suction play an important part in improving mortar compressive strength. The storage in envelopes for 21 days raised the strength only about 19 per cent. In another set of mortars stored loose on the shelf for 21 days; an apparent loss in strength was observed. Strengths of mortars made with dry hydrate averaged 1.11 times the corresponding values of mortars containing lime putties, which was close to the reciprocal of the ratios of the mortar and the masonry unit is incomplete, especially at an edge stressed in tension. Heretofore, lack of any satisfactory method of determining the area of contact of samples in the laboratory has hindered the development of a method of making up beams which would yield results of low scatter.

It has been a matter of observation in the field that areas where brick and mortar are not in contact become grimed.[10] A griming agent is needed for laboratory specimens, such as one of the "fugitive" or readily water-soluble dyes. A "blue dye"[11] seems to be satisfactory and can be applied in solution with a brush to a joint at an age of about 3 days. The solution should be so prepared as to have a high tinctorial value. Since greatest difficulty is experienced in getting complete contact all around the outside of the joint (the furrowed joint of the brick mason does not enter this particular discussion) this method readily indicates any noncontacting areas near the edge which is under tension during the loading.

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[10] Personal communication from C. C. Connor. Also F. O. Anderegg, "Watertight Brick Masonry," *Architectural Record*, p. 208 (1931), Figs. 8 and 10.

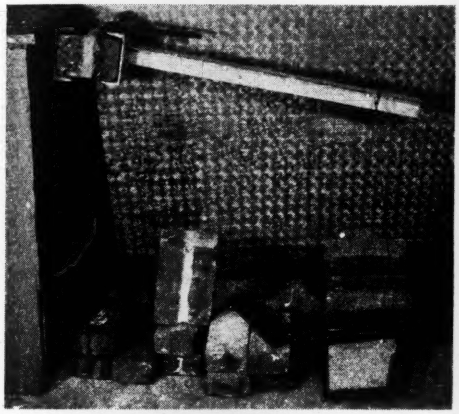

FIG. 2.—Breaking of 2-Brick Beams in Cantilever.

Later, as the technique improved, the channels were replaced by collars, one being bolted to a concrete wall, with a hydraulic cylinder for loading.

With the aid of this dye, a technique has been worked out for beam assembly that yields concordant results.[12] Just as in any method of testing cement products, meticulous attention to details is essential. The method is shown in Fig. 1, which illustrates a number of points. In the first place, the numbers painted on the brick are the weights in grams of water absorbed through one face in one minute. Brick are chosen having a narrow absorption range. In the second place, the consistency of the mortar must be controlled within a narrow range, which is chosen to fit the brick absorption, so that the surface of the brick will be adequately wetted. Not more than 3 beams may be made from one batch. The mortar must be applied to the brick surfaces with a wiping motion, so that contact is complete. Inserts may be used at the end of the joint to control the thickness of the joint. As soon as the bricks are superposed the upper one is pounded down until contact is made with the inserts. These inserts are removed very shortly and the spaces filled with fresh, soft mortar. Finally the joints are carefully tooled with a round tool, to be sure of complete contact all around between mortar and brick.

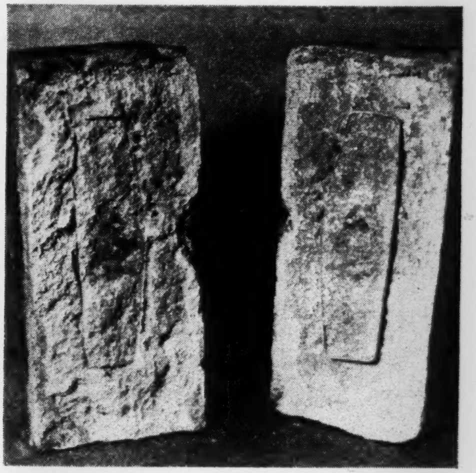

FIG. 3.—2:1:8 Mortar Bonds Broken at 28 days.

A large part of the area was still damp, especially when lime putty was used.

The method of breaking first used is shown in Fig. 2. Bond strengths at 28 days were generally too great for this apparatus and a new one, with collars bolted around the brick, was fabricated, the pressure being applied from an hydraulic jack.

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[11] The dye used was a "fugitive" dye for textiles from the Texply Co., Inc., Somerville, N. J.

[12] J. C. Pearson has also used two-brick beams but failed to get concordant results. Personal communication.

After breaking the beams in cantilever, the mortar was detached by sharp pounding on the brick. By including within the corner of the mortar a ¼-in. strip of thin oiled metal bent into a 1 by 6-in. rectangle, clipping of the mortar into suitable squares is facilitated. Such a frame may be seen directly under the trowl in Fig. 1 and again in Fig. 3, which shows a fracture. From 12 to 20 squares are tested from the mortar representing each combination of conditions. The results, after correcting to the cubical shape as described above, are given in Table III in the columns headed, "corrected square." The scatter among a given set is generally about ± 15 per cent. The results in the column headed "cube" were the compressive strengths of six cubes molded in metal from the same mortar placed in the joints. The scatter among these results was usually below 5 per cent.

Results are given with three cement-lime ratios and which have been given the literal designation *H*, *M*, and *N*, corresponding to designations proposed for different grades of mortar in preliminary discussions in A.S.T.M. Committee C-12 on Mortars for Unit Masonry.

TABLE III.—BOND AND COMPRESSIVE STRENGTHS OF MORTARS IN MASONRY.
(Values in pounds per square inch.)

Lime	Mix		7 Days			28 Days		
			Cube	Corrected square	Bond	Cube	Corrected square	Bond
B Dry Hydrate	*H*	2:1:8	2620	2500	147	4075	4490	318
	M	1:1:6	1270	1520	114	1600	2720	290
	N	1:2:8	450	1390	97	725	2320	214
	H/N	.....	2.96	1.80	1.52	5.62	1.90	1.49
B Putty	*H*	2:1:8	2100	3230	226	2670	4310	420
	M	1:1:6	1220	1670	116	1470	2560	243
	N	1:2:8	710	1150	74	880	1650	196
	H/N	.....	2.96	2.80	3.05	3.04	2.61	2.30
D Dry Hydrate	*H*	2:1:8	2370	2450	112	2950	4700	244
	M	1:1:6	750	1190	76	980	2000	192
	N	1:2:8	380	830	56	680	1230	160
	H/N	.....	6.24	2.95	2.00	4.34	3.82	1.53
E Dry Hydrate	*H*	2:1:8	3110	2820	149	3750	3670	261
	M	1:1:6	1140	1400	86	1225	1660	207
	N	1:2:8	670	1100	70	730	1670	152
	H/N	.....	4.65	2.38	2.13	5.13	2.20	1.72
F Putty	*H*	2:1:8	1440	2010	129	2400	3420	333
	M	1:1:6	510	1950	63	800	2070	159
	N	1:2:8	250	615	43	570	1380	96
	H/N	.....	5.76	3.27	3.00	4.22	2.44	3.47
Average	*H/N*	.....	5.09	2.64	2.34	4.65	2.40	2.00
Portland cement	....	1:3	2340	2520	280	3900	3700[a]	355
Average of 5 limes	....	1:3	84	None[b]	12	100	184[b]	27

[a] Squares cut from moist inside averaged 5180 psi. Those cut from dry outer part averaged 2220 psi.
[b] Suitable specimens unobtainable at 7 days from the mortar joint and only a few at 28 days.

Conclusions:

Bond strengths of low scatter have been obtained by sufficient attention to details, among which are the following: selection of brick of narrow absorption range; adjustment of mortar consistency to a flow of 110 per cent at the age of 1 hr. to insure wetting the surface of the brick; application of the mortar to the brick surface with a wiping motion; pounding down of the brick to the ½-in.

joint thickness; use within 10 min. of a batch of mortar; cementing together of only two bricks for a specimen beam; final tooling of the joint to force the mortar into complete contact with all brick surfaces; and use of a dye solution to determine the degree of contact.

The results obtained by this method and recorded in Table III seem sufficiently good to justify suggesting study of this method for an acceptance test for mortars. A minimum modulus of rupture of 50 psi. at 28 days, with joints essentially free of dye penetration, would be a suitable specification.

These bond strengths correlate reasonably well with the compressive strengths of mortar squares taken from between the brick but not with the cube strengths. The combined effect of packing and suction apparently cannot be emulated with metal molds.

Mortars high in portland cement seem to benefit much less from the packing-suction than do mortars higher in lime. This is one reason why the load-carrying capacity of masonry assemblies correlates so poorly with cube strengths.

The sand has an important effect on the results obtained. In this series, a silica sand of fairly good grading has yielded strengths higher than can usually be expected. It is important in the design of a given mortar mix to use not only the masonry units specified, but also the sand or sands available.

The preparation of the squares for crushing probably involves too much skill and labor to warrant inclusion in ordinary acceptance tests. It may be sufficient for most purposes to multiply the modulus of rupture, obtained at 28 days with joints essentially free of dye penetration, by a factor worked out experimentally for a given sand to give an estimate of the compressive strength of mortar within the brick for 28-day cube-shaped specimens. The ratio of the crushing strength of these silica-sand-mortar squares to the bond strengths averaged 11.2 ± 2.3, which is a reasonably low average scatter, everything considered.

In the 1:1:6 and 1:2:8 mixes, hydrated limes, because of lower water content in the mortar, yield the higher joint strengths, but in the 2:1:8 mixes, one of the lime putties has developed higher bond strengths than has the dry hydrate. The explanation is that the presence of the extra water held by the putty aids in the hydration of the portland cement. This is confirmed by the appearance of the joints after fracture. They were still moist at 28 days, as shown in Fig. 3.

The bond strengths and compressive strengths of squares in this series seem to be inversely proportional to the mortar yields. The cube strengths, however, seem to vary inversely as some power of the mortar yields. The effect of curing moisture was shown by the difference between the crushing strength of 1:3 cement mortar taken from the outside and inside of joints.

DURABILITY

Mortars made from local sand were adjusted after 10-min. mixing and 50-min. standing to a flow of 105 per cent, and were placed between bricks having a 1-min. absorption of 20 ± 4 g., with thin tissue paper between the mortar and the brick, the joints being pounded down to ½-in. thickness. After curing for one week between bricks, the slabs were removed, soaked for 24 hr. in water, and allowed to dry for 6 days. Samples of each mix were then subjected to one of the following tests:

(*a*) Freezing and thawing in a cabinet for 50 cycles. The cycle was 22 hr. freezing at 10 F. and 2 hr. thawing in warm water.

(*b*) Freezing and thawing outdoors and indoors for 15 cycles, the pan being brought in each morning and thawed out over the stove.

(*c*) One series of 14-day old specimens was soaked for 24 hr. and then placed, half immersed, in a pan of snow on the roof, about the middle of January, 1940. Observations were made early in the months of February, March, and April.

TABLE IV.—DURABILITY OF MORTAR FABRICATED BETWEEN BRICKS.[a]

Lime	Mix	Mixing Water, per cent	Cabinet Freezing and Thawing	Outdoors Freezing and Thawing	Left on Roof 3 Months	Sodium Sulfate Solution 5 Weeks
B Dry Hydrate	1:¼:3	21.1	O.K.	O.K.	O.K.	failed, 5th
	1:1:6	22.2	O.K.	O.K.	O.K.	failed, 5th
	1:2:8	23.6	90% left	failed, 9th	failed, 3d	failed, 3rd
B Putty	1:¼:3	24.2	O.K.	O.K.	O.K.	failed, 5th
	1:1:6	26.4	95% left	O.K.	O.K.	failed, 5th
	1:2:8	27.3	90% left	failed, 9th	failed, 3d	failed, 4th
D Dry Hydrate	1:¼:3	23.1	O.K.	O.K.	O.K.	failed, 5th
	1:1:6	....	O.K.	O.K.	O.K.	failed, 5th
	1:2:8	....	failed, 8th	O.K.	O.K.	failed, 5th
E Dry Hydrate	1:¼:3	21.1	O.K.	O.K.	O.K.	failed, 5th
	1:1:6	24.6	98% left	O.K.	O.K.	failed, 5th
	1:2:8	25.6	85% left	75% left	Ex.[b]	failed, 5th
F Putty	1:¼:3	24.3	O.K.	O.K.	O.K.	failed, 2nd
	1:1:6	29.2	90% left	85% left	O.K.	failed, 3d
	1:2:8	30.5	75% left	failed, 10th	failed, 3d	failed, 2nd
Masonry Cements						
A	1:3	....	failed, 8th	failed, 9th	failed, 2nd	failed, 2nd
B	1:3	....	failed, 20th	failed, 11th	90% left	failed, 6th
C	1:3	....	failed, 35th	failed, 11th	35% left	failed, 5th
Portland Cement						
A	1:3	....	O.K.	O.K.	O.K.	failed, 6th

[a] The expression, "95% left," means a loss of 5 per cent under the given exposure. "Failed, 9th" means disintegration complete at the ninth cycle or observation.

[b] Exfoliated at the top to the extent of about 10 per cent. The tops of these slabs were white on April 2 from calcium ulfate efflorescence.

Another set was placed in a bucket containing 10 per cent sodium sulfate solution. The bucket was insulated with 4 in. of Fiberglas wool and placed outdoors. Once a week it was brought in and the crystals were melted over the stove. The specimens were taken out, disintegrated mortar was removed, and the specimens were baked for 4 hr. at 110 C. Then they were replaced in the solution and returned outdoors. The excellent insulation and long, slow, cooling period permitted the growth of large crystals within the slabs, causing rapid disintegration. The results are given in Table IV.

Conclusions:

The first three methods of test seem to be of approximately equal severity. The simplest way to test would be to expose the specimens partially immersed in a pan outdoors through one winter. Sufficient sulfuric acid occurs in the winter rain water in the northeastern quarter of this country to give immersed mortar slabs in one winter season an exposure approximating that which a parapet wall might be expected to receive in about five years. The sodium sulfate test is too severe to evaluate mortars properly.

Specimens made from lime putty suffered more than those made from the same proportion of dry hydrate. Dolomitic and high-calcium mortars with similar water content seemed to be nearly on a par in these experiments.

Mortars containing more portland cement were more resistant. Masonry cement *B* in the sodium sulfate test resisted about as well as the portland cement specimens. This cement, having a slag base, would not be attacked chemically as severely as portland cement, thereby compensating for its much lower strength. It is believed that low permeability or absorption and strength are the most important factors in resisting disintegration.

The next set of experiments will include mortars fabricated in metal molds, mortars left between brick, and as many different mortars of varying age as can readily be obtained.

Finally, the philosophy of testing—that all materials should be tested under conditions as closely simulative of actual application as practicable—is believed to be borne out by the results given in this paper on masonry mortars, and the list of tests of mortar in actual contact with masonry units should be expanded.

DISCUSSION

Mr. James R. Withrow.[1]—I presume that experimental precautions have been taken against such possible criticism that when the steel bands, shown in Fig. 1, are inserted in the mortar they do not interrupt any natural effect. Mr. Anderegg drew the conclusion that the major effect was toward the absorbing material. Perhaps that is true, but there should be some way found, if it has not been found, of making certain that there is no lateral effect due to extrusion and evaporation from the side. That may be unimportant in the case of lime mortars, because of the carbonating effect.

Mr. F. O. Anderegg.[2]—The beams were broken in cantilever, so that the condition of the mortar in the joints near the neutral axis did not affect the result. The effect of the band on evaporation and mortar strength was repeatedly investigated by comparing the crushing strength of squares clipped from the outside with similar ones clipped from the inside. No difference was detectable in the case of mortars containing a reasonable amount of lime; but straight portland cement mortar from outside the band had less than half the compressive strength of the mortar from the inside of the band, as stated in Table III. The poor water-retaining capacity of portland cement mortars seems also to apply to loss of moisture by evaporation. This seems to indicate the desirability of having present sufficient finely divided material to help hold the moisture. The replacement of one third of portland cement by lime putty *B* appreciably increased the 28-day bond strength over straight portland cement mortar, by affording better curing conditions for the cement.

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[2] Consulting Specialist on Building Materials, Newark, Ohio.

RELATION OF WATER ABSORPTION AND STRENGTH OF BRICK TO ABRASIVE RESISTANCE

By J. W. McBurney,[1] R. H. Brink,[1] and A. R. Eberle[1]

Synopsis

The Kessler portable abrasion tester was used to determine the comparative abrasive resistance of approximately 50 clay, shale and fireclay bricks of known strength and absorption. These bricks were selected to represent the maximum range in properties commercially available. The results indicate that the abrasive resistance increases with increasing compressive strength and decreases with increasing absorption.

That the abrasive resistance of brick is related to their strength and water absorption was recognized in the Standard Specifications for Clay Sewer Brick[2] promulgated by the Society in 1924. In this specification bricks were classified into four grades on the basis of absorption, and compressive and transverse strength. These grades were recommended as suitable for resisting the abrasive action of sewage at various velocities.

As a preliminary to revising this specification, it appeared desirable to study the relations between abrasive resistance and absorption and strength. While a number of tests have been proposed for measuring abrasive resistance of ceramic materials, in but few cases have these tests been related to behavior in service. So far as the authors know, no tests have been compared with the action of sewage as an abrasive agent. In the absence of any laboratory test known to be related to service conditions of sewer brick, an arbitrary selection of a test method involving the use of a portable abrasion apparatus designed by D. W. Kessler was made on the basis of convenience. The evaluation of this test awaits the collection of further service data of the kind reported by Plummer.[3]

Source and Description of Test Specimens

The four bricks reported in Table I were selected to provide low, medium and high resistance to abrasion. Brick No. 1 is another specimen of the grade of brick represented by No. 1 in Table II and by No. 2 in Table III. Brick No. 2 corresponds to No. 1 of Table III. Bricks Nos. 3 and 4 both exceeded 18,000 psi. in compressive strength and had water absorptions by 5-hr. boiling of 3.1 and 1.8 per cent, respectively. They are vitrified and are made specially for use in sewer construction.

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[1] Senior Technologist, Junior Engineer, and Research Associate of the Structural Clay Products Institute, respectively, National Bureau of Standards, Washington, D C.

[2] Standard Specifications for Clay Sewer Brick (C 32 - 24), 1936 Book of A.S.T.M. Standards, Part II, p. 134.

[3] Harry C. Plummer, "Abrasive Action of Sewage on Brick Sewers," *Bulletin*, Am. Ceramic Soc., Vol. 16, No. 9, pp. 365-368 (1938).

The brick specimens described in Table II are the identical specimens listed in Table I of the paper on water absorption of building brick published as an appendix to the 1936 report of A.S.T.M. Committee C-3 on Brick.[4]

The following quotation from that paper refers to these specimens: "Records of the tests on 3886 bricks tested in the 1930 survey (see *Proceedings,*

TABLE I.—REPRODUCIBILITY OF ABRASION TESTS ON BRICK.

Brick	Face	Test	Loss, g.				Average Loss, g. per min.						
			1 min.	3 min.	5 min.	10 min.	1 min.	3 min.	5 min.	10 min.	Second and third min.	Fourth and fifth min.	Sixth to tenth min.
No. 1..........	Front	No. 1	3.30	6.49	9.71	15.28	3.30	2.16	1.94	1.53	1.60	1.61	1.12
		No. 2	3.48	7.24	[a]	....	3.48	2.41	[a]	....	1.88	[a]	....
		No. 3	5.20	10.85	16.15	[b]	5.20	3.62	3.23	[b]	2.84	2.65	[b]
	Back	No. 4	5.31	11.42	16.64	[b]	5.31	3.81	3.33	[b]	3.05	2.61	[b]
		No. 5	5.18	9.97	14.72	23.33	5.18	3.32	2.94	2.33	2.40	2.38	1.72
		No. 6	3.05	7.32	11.38	19.12	3.05	2.44	2.28	1.91	2.14	2.03	1.55
No. 2..........	Front	No. 1	0.32	0.95	1.58	3.10	0.32	0.32	0.32	0.31	0.32	0.32	0.30
		No. 2	0.37	0.99	1.65	3.24	0.37	0.33	0.33	0.32	0.31	0.33	0.32
		No. 3	0.36	0.97	1.66	3.30	0.36	0.32	0.33	0.33	0.30	0.35	0.33
	Back	No. 4	0.57	1.46	2.26	4.27	0.57	0.49	0.45	0.43	0.45	0.40	0.40
		No. 5	0.62	1.60	2.44	4.25	0.62	0.53	0.49	0.42	0.49	0.42	0.36
		No. 6	0.45	1.39	2.17	3.95	0.45	0.46	0.43	0.40	0.47	0.39	0.36
No. 3..........	Front	No. 1	....	....	0.65	1.34	....	....	0.13	0.13	....	....	0.14
No. 4..........	Front	No. 1	....	....	0.60	1.26	....	....	0.12	0.13	....	....	0.13

[a] Break from side of hole.
[b] Foot had reached limit of vertical motion before 10 min.

TABLE II.—ABRASIVE LOSS, STRENGTH AND WATER ABSORPTION OF BRICKS SELECTED FROM THE 1930 SURVEY.

Description of Bricks			Results of Tests					
Specimen[a]	Source	Method of Forming and Raw Material[b]	Abrasive Loss, g.		Compressive Strength, Flatwise, psi.	Modulus of Rupture, psi.	Water Absorption, per cent	
			Test A	Test B			24-hr. cold	5-hr. boiling
No. 1.............	Wisconsin	SC, C	19.7	19.5	1 830	512	25.6	31.3
No. 2.............	Detroit	SM, C	6.3	7.1	3 250	540	22.4	26.4
No. 3.............	Hudson Valley	SM, C	4.4	3.4	5 840	712	15.3	19.0
No. 4.............	New Hampshire	SM, C	4.2	3.5	7 820	1185	15.3	18.0
No. 5.............	Texas	DP, S	1.2	1.6	8 970	1100	16.1	18.2
No. 6.............	Colorado	DP, S	7.3	8.1	4 900	784	14.5	17.5
No. 7.............	Wyoming	DP, S	9.6	9.1	4 420	272	12.7	16.0
No. 8.............	Colorado	SC, S	1.6	2.1	6 840	1520	12.5	14.7
No. 9.............	Philadelphia	EC, C	1.5	5.0	5 980	755	8.1	12.2
No. 10.............	Cleveland	SC, S	1.0	4.7	10 330	650	8.2	10.3
No. 12.............	Alabama	DP, S	1.0	1.3	15 650	1890	4.0	6.7
No. 13.............	Detroit	SM, C	1.2	0.9	8 960	2800	2.8	3.3
No. 14.............	Illinois	SC, S	0.6	0.6	17 250	2745	0.6	2.6
No. 15.............	Ohio	SC, MCS	0.9	0.9	20 000	2610	0.2	0.7
No. 16.............	Baltimore	SC, C	0.7	0.7	12 280	2780	0.8	2.2
No. 17.............	New Hampshire	SM, C	0.8	0.8	8 670	2780	1.5	2.2
No. 19.............	Indiana	SC, MCS	0.5	0.6	12 500	2980	0.3	0.9
No. 21.............	Wisconsin	SM, C	0.5	0.4	15 410	....	0.4	0.5
No. 22.............	Ohio	SM, C	0.8	0.7	17 550	3250	0.1	0.2

[a] These numbers correspond to those in Table I of the 1936 paper on Water Absorption. According to the absorptions as originally determined, these numbers were in order of decreasing absorption by 5-hr. boiling.

[b] SM = soft mud. SC = side cut. EC = end cut.
C = clay. S = shale. MCS = mixture of clay and shale.
FC = fire clay. D = deaired. DP = dry press.

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[4] J. W. McBurney, "Water Absorption of Building Brick," *Proceedings,* Am. Soc. Testing Mats., Vol. 36, Part I, p. 260 (1936).

Am. Soc. Testing Mats., Vol. 33, Part II, p. 636 (1933)) were examined and 22 half-brick specimens were selected for test in the present investigation according to the following criteria: (1) the specimens should represent the maximum range in absorption; (2) all commercial methods of forming should be represented; (3) a variety of raw materials should be selected; and (4) the principal producing districts should be represented." In the present study no abrasion tests were made on bricks Nos. 11, 18 and 20 because of their irregular shape. The absorption values are the results of test No. 9 reported in the 1936 paper.[4] The same half bricks were used for both tests A and B and, therefore, the strength and absorption data presented in Table II apply to both tests.

TABLE III.—ABRASIVE LOSS, STRENGTH AND WATER ABSORPTION OF BRICKS SELECTED FROM THE 1936 EXPOSURE TEST.

Description of Bricks			Results of Tests									
Specimen[a]	Locality	Method of Forming and Raw Materials	Abrasive Loss, g.		Compressive Strength, Flatwise, psi.		Modulus of Rupture, psi.		Water Absorption, per cent			
									24-hr. cold		5-hr. boiling	
			Test C	Test D	Test C	Test D	Test C	Test D	Test C	Test D	Test C	Test D
No. 1........	Texas	DP, C	6.4	....	3 230	....	775	....	22.7	....	31.0	....
No. 2........	Wisconsin	SC, C	5.9	....	4 245	....	1160	....	23.0	....	26.0	....
No. 3........	Pennsylvania	SM, C	27.2	25.6	1 440	1 265	195	216	16.3	16.7	24.0	23.7
No. 4........	Hudson Valley	SM, C	18.8	8.7	1 580	2 460	245	218	22.1	22.3	24.0	24.3
No. 5........	Detroit	SM, C	5.9	7.5	4 130	3 680	625	825	19.9	21.1	21.9	23.9
No. 6........	Hudson Valley	SM, C	7.5	5.6	4 350	3 960	595	1120	17.7	16.6	21.2	21.3
No. 7........	New England	SM, C	6.4	4.0	5 000	5 400	600	850	15.8	13.6	18.2	16.8
No. 8........	Philadelphia	EC, C	4.5	3.4	5 590	6 420	770	860	12.7	12.3	17.8	17.3
No. 9........	Pennsylvania	SM, C	2.5	....	5 670	....	1085	....	11.6	....	16.3	....
No. 10........	Chicago	EC, C	2.0	....	3 650	....	1660	....	12.1	....	15.9	....
No. 11........	New England	SM, C	3.2	2.5	9 200	6 570	1365	665	13.2	14.1	15.6	17.3
No. 12........	Virginia	DP, C	9.2	5.8	3 920	3 900	405	915	12.7	10.8	15.1	13.5
No. 13........	Texas	DP, S	2.2	1.9	7 370	6 540	775	810	12.8	12.7	14.3	14.2
No. 14........	Maryland	SC, C	5.7	4.5	4 190	4 720	335	471	9.6	9.1	12.8	12.5
No. 15........	West Virginia	SC, S	1.7	....	5 670	....	1495	....	10.2	....	11.8	....
No. 16........	Baltimore	SC, C	1.3	1.9	9 230	6 790	1000	762	9.8	10.7	11.5	12.7
No 17........	Pennsylvania	SC, S, D	2.3	....	6 540	....	540	....	9.4	....	11.0	....
No. 18........	Iowa	SC, S, D	1.1	0.9	16 330	9 570	820	700	3.2	2.4	8.8	10.7
No. 19........	New England	SM, C	1.0	0.8	11 600	12 140	1364	1725	4.6	4.9	7.3	8.1
No. 20........	New England	SM, C	0.4	0.6	13 500	14 200	1170	1690	3.8	1.4	5.7	2.3
No. 21........	West Virginia	SC, S	0.9	0.7	11 750	12 700	2015	2605	3.2	2.3	5.5	4.2
No. 22........	Pennsylvania	SC, FC	0.1	0.6	14 730	13 780	2335	2930	0.7	0.7	2.2	2.0

[a] Bricks arranged in order of decreasing absorption by 5-hr. boiling of Test C.

TABLE IV.—ABRASIVE LOSS, STRENGTH AND WATER ABSORPTION OF A SERIES OF DRY PRESS BRICKS FROM ST. LOUIS.

Specimen[a]	Abrasive Loss, Test E, g.	Compressive Strength, Flatwise, psi.	Modulus of Rupture, psi.	Water Absorption, per cent	
				24-hr. cold	5-hr. boiling
No. 1.......	5.3	5 655	680	13.9	16.4
No. 2.......	4.2	6 550	785	12.6	15.1
No. 3.......	2.6	6 935	645	11.5	14.1
No. 4.......	2.9	8 135	820	11.2	13.6
No. 5.......	3.0	8 515	515	10.9	13.3
No. 6.......	1.7	13 300	1105	8.8	11.3
No. 7.......	1.8	12 300	1285	8.8	11.3
No. 8.......	1.8	10 300	850	8.6	11.0
No. 9.......	1.5	14 700	1280	8.3	10.9
No. 10.......	1.2	14 200	1130	7.7	10.2

[a] Bricks arranged in order of decreasing absorption by 5-hr. boiling.

The brick specimens reported in Table III are samples of the same series of bricks which had been selected to undergo weathering exposure in the Bureau's "cemetery" exposure plot described in the paper on the wick test.[5] Since abrasion tests C and D were made on separate specimens, the individual strength and absorption values for these specimens are given in Table III.

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[5] J. W. McBurney and D. E. Parsons, "The Wick Test for Efflorescence of Building Brick," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 332 (1937).

Table IV presents data on a series of dry-press bricks from the St. Louis district characterized by both high strength and relatively high water absorption.

The data reported in Table V were obtained on the same grades (but not the same specimens) of brick as those listed under the corresponding numbers in Table III.

Description of Apparatus and Methods of Test

Kessler Apparatus:

A portable abrasion machine (shown in Fig. 1) designed by Kessler was used. This should not be confused with the apparatus described by Kessler in 1928.[6] The portable machine consists essentially of a vertical, hollow, loaded shaft, the lower end of which is fitted with a disk-shape foot. The shaft with its attached foot is free to move vertically and is rotated by a motor. Abrasive (No. 60 corundum or silicon carbide) is supplied from the top through the hollow shaft and flows continuously between the rotating foot and the brick surface being abraded.

TABLE V.—COMPARISON OF ABRASIVE LOSS ON DRY AND ON WATER-SATURATED BRICKS SELECTED FROM THE 1936 EXPOSURE TEST.

Description of Bricks			Results of Tests					
			Abrasive Loss, g.		Strength		Water Absorption, per cent	
Specimen[a]	Locality	Method of Forming and Raw Materials	Test F, dry	Test G, wet	Compressive Strength, Flatwise, psi.	Modulus of Rupture, psi.	24-hr. cold	5-hr. boiling
No. 1	Texas	DP, C	6.6	9.7	2 500	285	29.4	35.5
No. 3	Pennsylvania	SM, C	33.4	31.0	1 400	240	16.1	24.5
No. 4	Hudson Valley	SM, C	15.6	19.6	1 930	265	22.4	25.5
No. 8	Philadelphia	EC, C	3.8	7.4	4 900	930	11.4	16.0
No. 13	Texas	DP, S	2.3	2.9	5 600	1230	12.2	14.7
No. 15	West Virginia	SC, S	2.2	2.1	6 500	1110	11.7	13.5
No. 21	West Virginia	SC, S	0.7	0.8	8 300	1620	2.6	4.5
No. 22	Pennsylvania	SC, FC	0.6	1.0	16 850	2260	1.0	3.0
Average			8.2	9.3				

[a] These numbers designate the same grades of the same manufacturers but not the same specimens as the corresponding numbers in Table III.

The foot used in tests A and B (Table I) and C (Table III) was $2\frac{1}{8}$ in. in diameter with a 1-in. diameter hole through the center. Four radial slots, $\frac{1}{4}$ in. wide, were cut in the lower face of the foot to allow distribution of the abrasive.

Tests D (Table III), E (Table IV) and F and G (Table V) were made with the foot illustrated in Fig. 2. It was thought that the eccentricity of the $1\frac{15}{64}$-in. hole would provide better distribution of the abrasive and hence improve the reproducibility of the tests, but no systematic difference in the results was observed. Neither did any significant differences appear in the action of No. 60 corundum (tests A, B and C) compared with No. 60 silicon carbide.

Abrasive Test by Dry Method:

The method of test consisted in supporting the brick on edge under the foot so that the upper face of the brick was level and provided a reasonably uniform bearing for the foot. Except in the case of test A, a preliminary grinding was conducted long enough to produce a smooth bearing surface before measurement was begun, after which the brick was cleaned by vigorous brushing, weighed to the nearest 0.01 g., and replaced under the foot. The machine was then operated for 5.0 min. at a speed of 100 r.p.m. with the abrasive flowing at 3 to 4 g. per min., and a total load of 4.0 kg. acting on the foot, and the resulting loss in weight of the brick was determined.

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[6] D. W. Kessler, "The Development of an Apparatus for Wear Tests on Flooring Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 855 (1928).

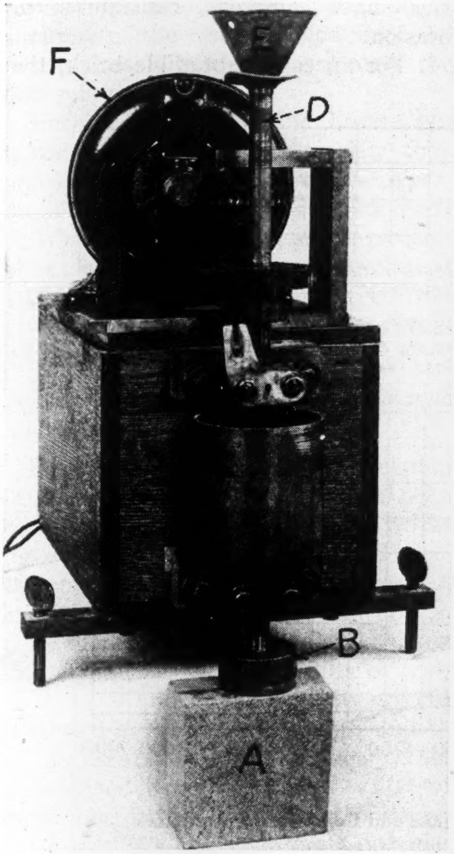

FIG. 1.—Kessler Portable Abrasion Testing Apparatus.

A specimen. *B* abrasion disk. *C* weight. *D* hollow shaft. *E* hopper for abrasive. *F* motor.

Abrasive Test by Wet Method:

Test G differed from tests A to F in that it was made on bricks saturated with water and totally immersed in water during the process of abrasion. Preliminary grinding of the face was done on the dry bricks as in tests B to F. The specimens were weighed, boiled in water for 5 hr., cooled overnight under water and then tested. During test the level of the water stood approximately $\frac{1}{8}$ in. above the top surface of the brick. The flow of abrasive for tests F and G was 4 to 5 g. per min. and in test G a small flow of water (5 ml. per min.) was used to wash the abrasive down the shaft. After abrasion the specimens were dried for 5 days at 120 C., brushed and reweighed.

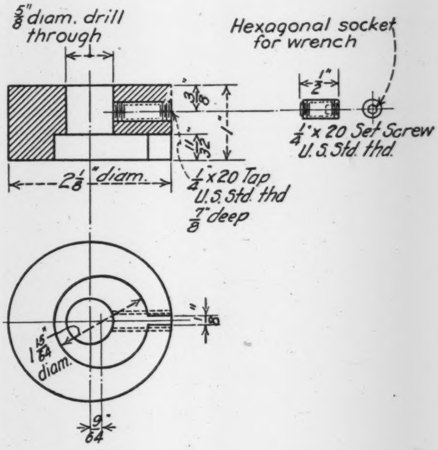
5/8" diam. drill through
Hexagonal socket for wrench
3/16
1"
1/2"
11/32
1/4" x 20 Set Screw U. S. Std. thd.
2 1/8" diam.
1/4" x 20 Tap U. S. Std. thd 7/8" deep
1 15/64" diam.
1/8
9/64"

FIG. 2.—Diagram of Abrading Disk with Eccentric Hole.

Special Abrasive Tests:

The data recorded in Table I were obtained by the following methods: On bricks Nos. 1 and 2, the grinding disk with concentric hole and radial slots was used. Bricks Nos. 3 and 4 were tested by use of the disk with eccentric hole (Fig. 2). No preliminary abrading of the faces of bricks Nos. 1 and 2 was done. On bricks Nos. 3 and 4 the apparatus was operated until a complete circular depression was worn on the specimen before taking the initial weight. Each of the tests Nos. 1 to 6 on bricks Nos. 1 and 2 was made on a different portion of the brick. After each abrading interval (1, 3, 5 and 10 min.) the specimen was cleaned and reweighed and replaced in the same position it previously occupied.

Strength and Absorption:

All strength and water-absorption measurements were made according to Standard Methods C 67 - 39 of the Society.[7]

Discussion of Data

Reproducibility of Data:

From the data presented in Table I, the following conclusions appear to be justified:

1. Different portions of the same brick have different resistances to abrasion.

2. For an easily abradible brick, the average rate of abrasion decreases with increasing duration of test period.

3. For a brick with a low abrasive loss, the rate of loss in grams per minute is little affected by duration of the test within the limits reported.

Conclusion 1 is explained by lack of homogeneity of the brick. The effect is particularly noticeable in bricks of low compressive strength since in such bricks small differences in strength are asso-

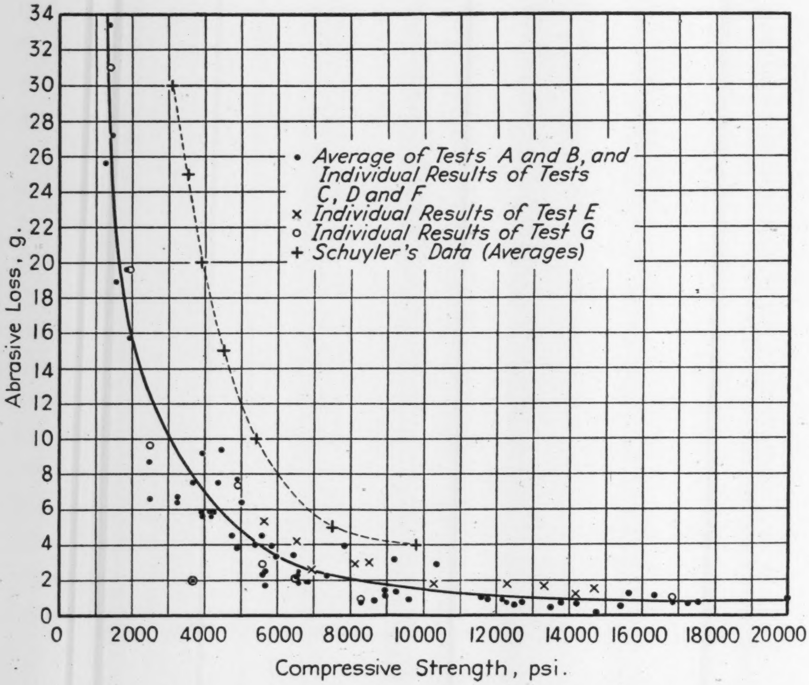

Fig. 3.—Relation Between Abrasive Loss and Compressive Strength.

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[7] Standard Methods of Sampling and Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (C67–39), 1939 Book of A.S.T.M. Standards, Part II, p. 104.

ciated with large differences in abradibility (see Fig. 3).

The obvious explanation of the phenomenon reported in conclusion 2 is that the abrasive becomes diluted with brick dust as the hole deepens. The data providing conclusions 2 and 3 also provide justification for a preliminary abrading of the test specimen since the rate of loss is more uniform after the first minute.

Since the data of Table I were obtained on a variable material, they provide little information on the reproducibility of the test. In six tests made on adjacent portions of a single piece of plate glass which gave an average loss of 1.23 g. in 15 min., it was observed that the range in abrasive results was within ±2.5 per cent of the average.

Relation of Abrasive Resistance to Strength of Bricks:

Figure 3 illustrates the relation between the abrasive loss and the compressive strength of the bricks reported in Tables II to V. In Fig. 3, the abrasive losses reported for tests A and B have been averaged for each brick. Since there appeared to be no systematic differences between the results of tests A, B, C, D and F, these results are not separated as are those of tests E and G.

The points tend to fall in a rather well-defined band following a curve of hyperbolic form. The circled point on the 2.0-g., 3650-psi. coordinate provides an exception to this statement. This point represents the test on a Chicago common brick which is peculiar in that its compressive strength tested endwise is much higher than when tested flatwise. The average strength of 25 of these bricks was 3910 psi. tested flatwise, and of a comparable sample of 25 similar bricks tested on end was 7030 psi. If this higher value for compressive strength were plotted against abrasive loss in Fig. 3, the resulting point would fall very close to the line representing the average relation.

Test E (Table IV) was made on a series of dry-press bricks having relatively high water absorptions in relation to their strength. The abrasive losses on these bricks were usually higher than those on bricks having similar compressive strengths but lower absorptions.

Test G (Table V) differed from all other tests in that the bricks were saturated with water before abrading and were tested under water. Although Fig. 3 indicates that the results of test G did not differ appreciably from those of tests A, B, C, D and F, a direct comparison of tests F and G (Table V) shows that in 6 out of 8 cases test G gave a greater abrasive loss. The only specimen (No. 3) which gave an appreciably greater loss with test F was also the most abradible of the series. Clogging of the wet abrasive may explain this reversal. The differences, however, in the results of the two methods do not, in general, appear to be important.

The average results from some of Schuyler's data,[8] obtained by use of a centrifugal sand blast, are shown in Fig. 3 for comparison with the data from the present investigation. Schuyler's data include, in addition to the results indicated in Fig. 3, a loss of 80 g. obtained on a brick of 1900-psi. compressive strength. Schuyler's method is obviously more severe than the method described in this paper. His curve is displaced in the manner that would result from increasing time or load with the Kessler machine, but the shape of the two curves is approximately the same. Both curves indicate that bricks exceeding 8000 psi. in compressive strength are relatively resistant to abrasive wear and that for bricks of low strength (less than 4500 psi.), small strength differences are associated with large differences in abrasive resistance.

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[8] Mont Schuyler, "A Machine for Testing Clay Products," *Proceedings*, Am. Soc. Testing Mats., Vol. XIV, Part II, p. 557 (1914).

Plotting modulus of rupture (Tables II to V) against abrasive loss would give a relation closely resembling that shown in Fig. 3, with the difference that the individual points show a wider scattering, as would be expected from the fact that moduli of rupture are affected by kiln cracks, laminar structure, and nodules.

Relation of Abrasive Resistance to Water Absorption of Bricks:

Figure 4 shows that the abrasive loss tended to increase with the amount of water absorbed by 5-hr. boiling. All bricks with absorptions less than 10 per cent lost less than 1.3 g. in the abrasion test. There is a small but definite trend toward lowered abrasive loss as absorption decreased from 10 per cent and approached zero. As the absorption increased above 10 per cent, the trend toward higher abrasive loss increased, but the scatter in results became very large. All losses exceeding 10 g. were obtained on brick exceeding 23.6 per cent in absorption.

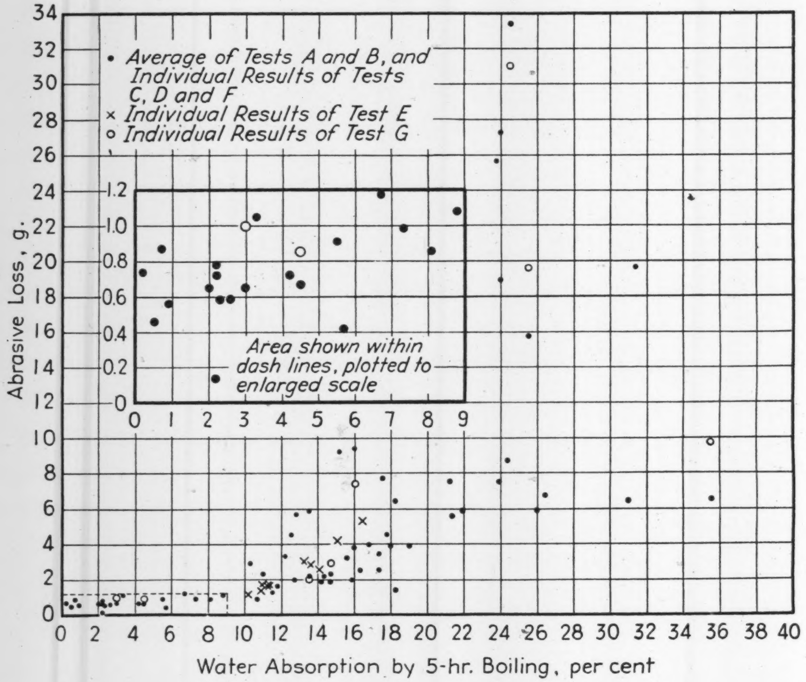

Fig. 4.—Relation Between Abrasive Loss and Water Absorption by 5-hr. Boiling.

It will be noted from Fig. 4 that the abrasive losses recorded for test E are about average in comparison with other bricks of the same absorption, while Fig. 3 shows that they had somewhat high abrasive loss in comparison with other bricks of the same strength. Both figures should be considered in predicting abrasive resistance from the other properties, since Fig. 3 fixes the abrasive loss within fairly close limits, but Fig. 4

enables us to apply a qualitative correction, which in the case of test E would suggest that these points in Fig. 3 would be above the curve.

The results of water absorption determination by 24-hr. cold immersion could be substituted for absorption by 5-hr. boiling in Fig. 4 without material change in the nature of the relation.

SUMMARY AND CONCLUSIONS

The Kessler portable abrasion tester was used to determine the comparative abrasive resistance of approximately 50 clay, fire-clay, and shale bricks of known strength and absorption. These bricks had been selected to represent the maximum range in properties commercially available. From the data here presented the following conclusions appear to be justified:

1. The abrasive resistance of bricks increases with their compressive strength. Plotting abrasive loss against compressive strength gave points which fall in a band of hyperbolic form.

2. The abrasive resistance of bricks decreases as water absorption increases. This relation is rather definite for bricks with absorption by 5-hr. boiling less than 10 per cent. Above 10 per cent the scatter becomes very large.

3. Under the conditions of test, abrasive losses determined on bricks saturated with water were not significantly greater than those determined on the same bricks tested dry.

DISCUSSION

Mr. H. C. Plummer[1] (*presented in written form*).—The data presented by Mr. McBurney have been of particular value to the Sewer Brick Subcommittee of Committee C-15 on Manufactured Masonry units in revising the Standard Specifications for Sewer Brick (C 32 – 24).[2]

Abrasive resistance has long been recognized as one of the principal requirements of sewer brick and while the data are meager as to the abrasive action of various types of sewage at different velocities, the consensus of those concerned with the problem has been for some time that the inverts of sewers, particularly those carrying sewage at fairly high velocities (8 ft. per second and over), should be constructed of materials having a high resistance to abrasion.

Specifications C 32 – 24 set up four grades of sewer brick: Class A Vitrified, with average absorption of 3 per cent or less; Class B Vitrified, with average absorption of 5 per cent or less; Hard, with average absorption of 5 to 10 per cent, and Medium, with average absorption of 10 to 15 per cent.

Presumably, the maximum absorption together with the minimum strength requirements were supposed to be a measure of the abrasive resistance as well as other desirable properties of the various grades, and it was assumed that there were significant differences in the abrasive resistance and other properties of each grade.

The data presented by Mr. McBurney indicate that there is a relation between absorption and abrasive resistance, and that, in general, bricks with low absorption have higher abrasive resistance than brick of higher absorption. However, as indicated by Fig. 4, this relationship is by no means linear, and for absorptions of 10 per cent and under, there seems to be only a slight increase in abrasive resistance with decrease in absorption by 5-hr. boil. For the specimens tested, the difference in abrasive loss between the bricks of 3 and 10 per cent absorption was only 0.5 g. Certainly the difference in abrasive resistance of 3 and 5 per cent absorption brick could hardly be considered significant.

In the new Tentative Specifications for Sewer Brick (Made from Clay or Shale) (C 32 – 39 T),[3] three grades of brick are included: *SA* with an average absorption of 6 per cent or less; *MA* with an average absorption of 12 per cent or less, and *NA* with an average absorption of 20 per cent or less. These specifications conform more nearly to Mr. McBurney's data in grading brick according to their abrasive resistance. However, since his data indicate that no losses exceeding 2 g. are associated with brick of absorption of less than 10 per cent, the average maximum of 6 per cent and individual maximum of 9 per cent for the top grade appear conservative. The data justify an average of at least 7 per cent and an individual maximum of 10 per cent.

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[1] Director of Engineering and Research, Structural Clay Products Inst., Washington, D. C.

[2] 1936 Book of A.S.T.M. Standards, Part II, p. 134.

[3] 1939 Book of A.S.T.M. Standards, Part II, p. 925.

The compressive strength requirement for "Class A Vitrified" brick in C 32 – 24 was an individual average and minimum of not less than 5000 psi. The "Class B Vitrified" brick had the same requirement for average strength but permitted an individual minimum of 4000 psi. At first glance, the compressive strength requirement for grade *SA* of C 32 – 39 T (average of 5 bricks no less than 8000 psi. and individual minimum not less than 5000 psi.) appear to be materially higher than the corresponding requirements in C 32 – 24. Actually the compressive strengths are not directly comparable because C 32 – 39 T requires testing flatwise while C 32 – 24 prescribes testing on edge. Bleininger[4] reported, on the basis of 176 tests, that the average ratio of compressive strength flat to compressive strength on edge was 1.153 and "individual tests vary widely from the average ratio." McBurney[5] gave no averages but concluded that "the ratio compressive strength of brick flat to compressive strength of brick on edge ranges from 0.74 to 2.3."

From Fig. 3 it appears that a minimum compressive strength of 11,000 psi. would correspond to the maximum of 10 per cent by 5-hr. boiling in terms of limiting abrasive loss to 2 g. That a compressive strength of 11,000 psi., like 10 per cent absorption, limits bricks to a 2-g. loss in weight may be chance. It is an interesting coincidence that tests on over 2100 bricks indicate that a minimum of 11,000 psi. for strength and a maximum of 10 per cent in absorption would be necessary to guarantee that no bricks lose more than 3 per cent in weight subjected to 50 cycles of freezing and thawing.

The average maximum absorption by 5-hr. boiling (12 per cent) prescribed for the *MA* grade limits abrasive loss to 4.0 g. The individual maximum (16 per cent) permits a 6.0-g. loss. The two specimens with absorptions less than 16 per cent which have abrasive losses exceeding 9.0 g., have compressive strengths less than 5000 psi. which is the corresponding average minimum for grade *MA*. The 10 per cent limit on absorption for the corresponding "hard" grade of C 32 – 24 may have been selected for the reason that shale bricks with absorptions above 10 per cent may be "underburned." A requirement of a maximum saturation coefficient (*C*/*B* ratio) of 0.80 would eliminate "underburned" shale brick and at the same time permit the use of certain clay bricks which do not require absorption limits as low as 10 per cent."

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[4] A. V. Bleininger, "The Relation Between the Porosity and Crushing Strength of Clay Products," *Transactions*, Am. Ceramic Soc., Vol. 12, pp. 564–584 (1910).

[5] J. W. McBurney, "The Compressive and Transverse Strength of Brick," *Journal of Research*, Nat. Bureau Standards, Vol. 2, April, 1939, pp. 821–835 (*R.P.* 59).

THE MECHANICS OF GRANULAR AND GRANULAR-PLASTIC MATERIALS, WITH SPECIAL REFERENCE TO BITUMINOUS ROAD MATERIALS AND SUBSOILS

By V. A. Endersby[1]

Synopsis

The object of the investigation was to separate, more definitely than heretofore, the respective functions of aggregate and binder in bituminous mixes. The method followed was the triaxial test, with study of void changes. Results were evaluated by the Mohr circle diagram. A means was found for separating certain nonfrictional and noncohesive resistances of a primarily dimensional nature from values of apparent friction and apparent cohesion, thus leading to a clarification of the conceptions of granular-plastic action, a method of evaluating the errors involved in applying the classic formulas to conditions where dimensional effects exist, and a method of determining the functions of bitumen *versus* aggregate in triaxial road mix samples of various height diameter ratios.

It was found that in order to correlate tests with practical conditions, the absolute dimensions of the specimen, its proportions, the maximum particle size, the temperature, and the speed, must all be considered. These same phenomena need study in soil mechanics as well as in bituminous road work, for the purposes of securing consistency of results between investigators and correlating theory with practical application.

The investigations described here were undertaken for the purpose of analyzing the contribution of asphalt to the mechanical stability of mixes, this being a principal one of its complex functions in the pavement. Such analysis can only be made by means that are capable of measuring all forces acting within the complete system.

The various forms of resistance to external forces, presented by matter in general, may be elastic, frictional, viscous, or plastic, or any combination of these. In road materials as a class, resistance is considered to be frictional in noncohesive materials, and plastic in cohesive materials. This form of plasticity is due to the combined effect of internal friction and internal cohesion. In cases where the cohesive agent is very resistant, elastic phenomena are found, but with bituminous binders, except under conditions of high loading speed, low temperature, or very hard binder, the phenomena are essentially plastic, in the sense that no spontaneous restoration of form of a deformed specimen occurs upon removal of load. Since, with viscous materials, such as asphalt, the least resistance to shear occurs at the slowest loading speed, this condition represents the logical approach to a study of shearing phenomena. In the studies described, a temperature of 77 F.

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[1] Research Engineer, Shell Development Co., Emeryville, Calif.

was employed, because it is easy to use, probably represents a good year-round average, and brings out the role of the binder more clearly than does a higher temperature.

MECHANICS AND MATHEMATICAL BASIS OF EXPERIMENTAL METHODS USED

A body of material in a pavement is subjected to a combination of vertical load and lateral restraint. Under certain conditions, tensile and flexural phenomena become important, but are not dealt with here. Experimental means of measuring the effect of this combination are called for. In addition, an analysis of the respective functions of binder and aggregate require a method which will show the cohesion and friction as separate forces. All these conditions are fulfilled by the triaxial test when combined with the Mohr diagram type of representation of results.

The triaxial test is a compression test made upon a cylindrical specimen to whose sides are applied hydraulic pressure. In our work this is carried out by enclosing the specimen in a rubber tube. Details of the equipment will be found in Fig. 1, while Fig. 2 shows a general view.

With a cohesive specimen it is only necessary to slide it into the tube, unfastening one of the clamps after the test in order to remove the deformed material.

A noncohesive specimen is vibrated to compaction in the rubber tube, which remains mounted on its base, and is enclosed in a split mold. Inside the mold

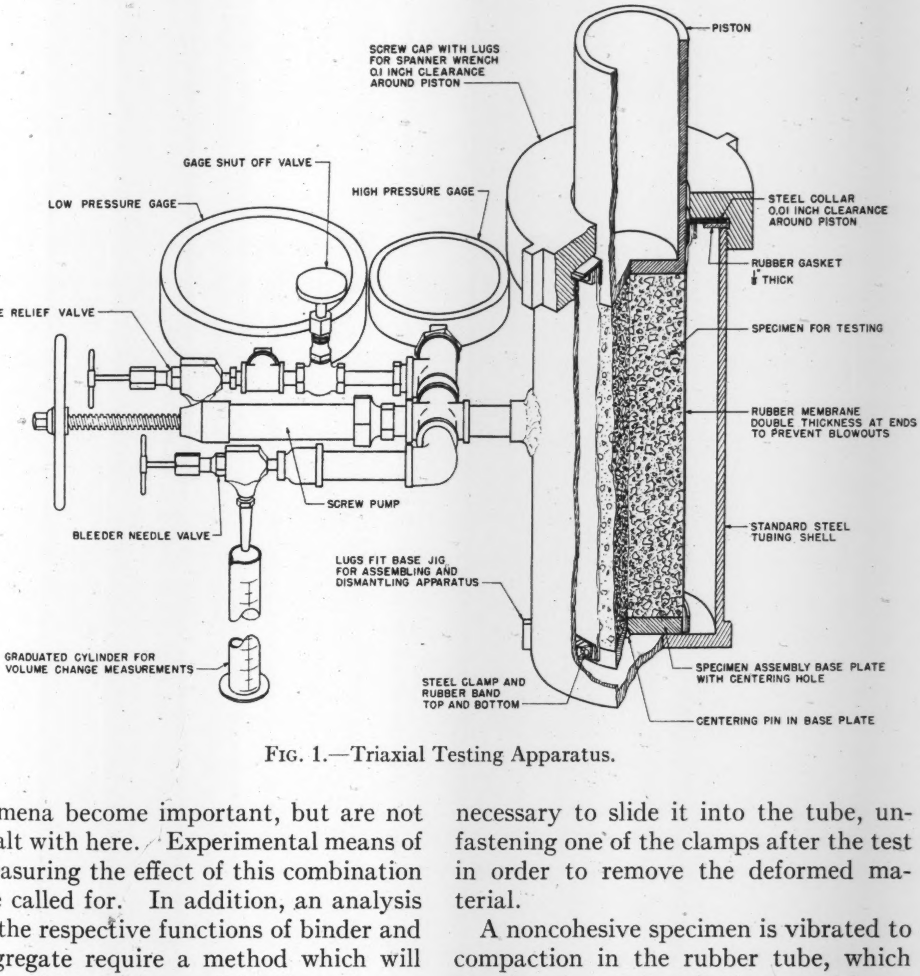
PISTON
SCREW CAP WITH LUGS FOR SPANNER WRENCH 0.1 INCH CLEARANCE AROUND PISTON
GAGE SHUT OFF VALVE
HIGH PRESSURE GAGE
LOW PRESSURE GAGE
STEEL COLLAR 0.01 INCH CLEARANCE AROUND PISTON
RUBBER GASKET ⅛" THICK
GAGE RELIEF VALVE
SPECIMEN FOR TESTING
RUBBER MEMBRANE DOUBLE THICKNESS AT ENDS TO PREVENT BLOWOUTS
SCREW PUMP
STANDARD STEEL TUBING SHELL
BLEEDER NEEDLE VALVE
LUGS FIT BASE JIG FOR ASSEMBLING AND DISMANTLING APPARATUS
GRADUATED CYLINDER FOR VOLUME CHANGE MEASUREMENTS
STEEL CLAMP AND RUBBER BAND TOP AND BOTTOM
SPECIMEN ASSEMBLY BASE PLATE WITH CENTERING HOLE
CENTERING PIN IN BASE PLATE

FIG. 1.—Triaxial Testing Apparatus.

and around the tube is a paper sleeve fastened with water-soluble glue. This is left in place to maintain the diameter until the specimen in its tube is placed in the outer cylinder. The paper falls off when the glue is softened by the surrounding water.

With both cohesive and noncohesive materials, the plunger of the apparatus is set carefully upon the specimen, the outer cylinder is filled with water, care being used to exclude bubbles, and the cap of the outer cylinder is screwed down, a valve being left open to prevent generation of pressure as the gasket compresses. The assembly is then placed in the testing machine, the head of the machine brought carefully into contact with the plunger, and pressure is applied by the screw pump, after which vertical load is applied at constant speed.

The following general methods of test are possible:

(*a*) It is assumed that the material is linear-plastic: that is, that under a constant rate of deformation all forces are constant and independent of extent of deformation. By this method a predetermined lateral pressure is applied and maintained by operation of the pump and bleeder valve, while the vertical loading is watched until it also becomes constant. The values are recorded, and the pressure is stepped up,

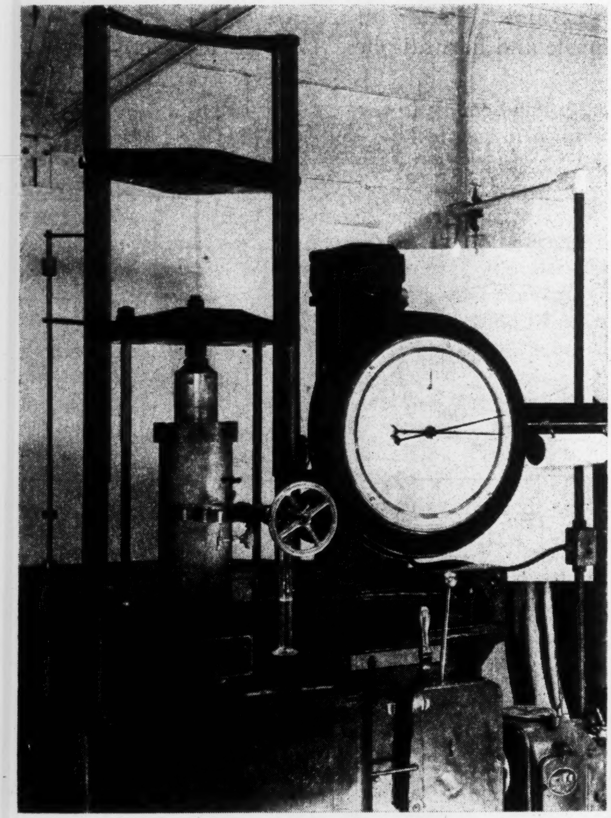

FIG. 2.—Emeryville Triaxial Apparatus in Position in Testing Machine.

the process being repeated as desired. At least three pressure-stages should be recorded. Alternatively, the test may start with a high pressure, which is gradually dropped; and it is merely a matter of convenience whether the vertical load or the lateral pressure is predetermined. For cohesive materials, the first stage should be at zero lateral load, which is equivalent to an ordinary compression test.

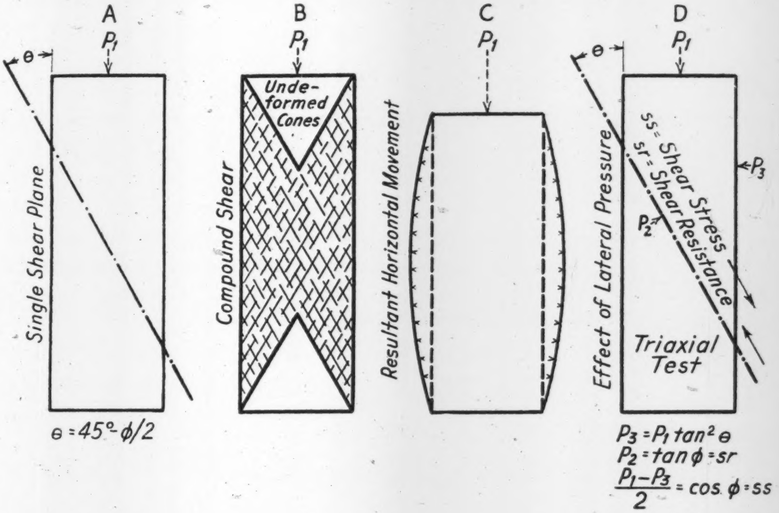
A
B
C
D
P_1
θ
Single Shear Plane
θ = 45° - φ/2
Undeformed Cones
Compound Shear
Resultant Horizontal Movement
Effect of Lateral Pressure
ss = Shear Stress
sr = Shear Resistance
P_3
P_2
Triaxial Test
$P_3 = P_1 \tan^2 \theta$
$P_2 = \tan \phi = sr$
$\frac{P_1 - P_3}{2} = \cos \phi = ss$

FIG. 3.—Fundamentals of Compression Failure.

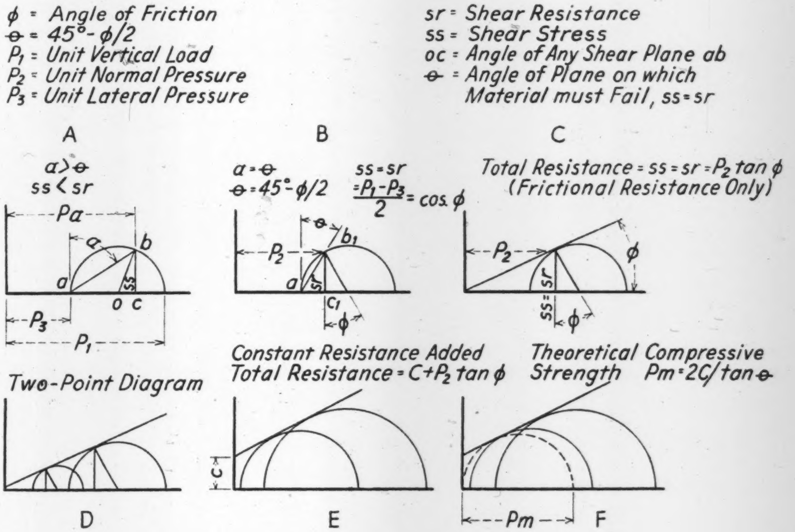
φ = Angle of Friction
θ = 45° - φ/2
P_1 = Unit Vertical Load
P_2 = Unit Normal Pressure
P_3 = Unit Lateral Pressure
sr = Shear Resistance
ss = Shear Stress
oc = Angle of Any Shear Plane ab
θ = Angle of Plane on which Material must Fail, ss = sr
A
a > θ
ss < sr
Pa
a
b
o c
ss
P_3
P_1
B
a = θ
θ = 45° - φ/2
ss = sr = $\frac{P_1 - P_3}{2}$ = cos φ
P_2
b_1
c_1
φ
C
Total Resistance = ss = sr = P_2 tan φ
(Frictional Resistance Only)
ss = sr
Two-Point Diagram
D
Constant Resistance Added
Total Resistance = C + P_2 tan φ
C
E
Theoretical Compressive Strength Pm = 2C/tan θ
Pm
F

FIG. 4.—Elements of the Mohr Diagram.

(*b*) A stress-strain curve is recorded for a new specimen at each stage of pressure; any number of Mohr diagrams can then be made, each corresponding to a different stage of strain, this stage being the same for each specimen for a given diagram. For instance, method (*a*) corresponds to that stage of the stress-strain curve where the peak is well past and the curve has flattened out, parallel with the strain axis.

In the Mohr diagrams following, method (*a*) is used for illustration. In most materials, however, a true linear-plastic condition can be only approximated, because of the extensive duration of void changes during deformation.

Where void changes are studied, the volume of water extruded through the bleeder valve is compared with the piston travel.

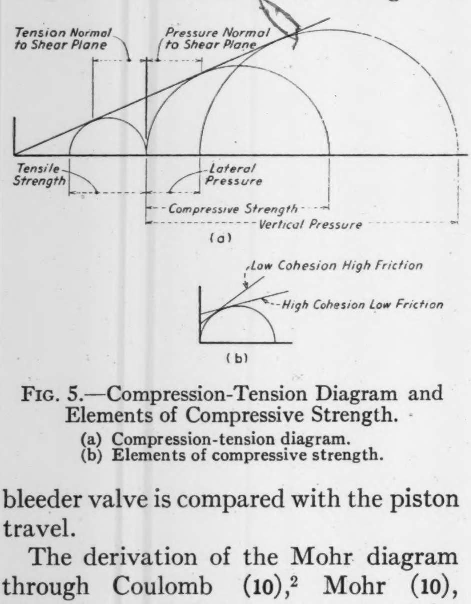
Tension Normal to Shear Plane
Pressure Normal to Shear Plane
Tensile Strength
Lateral Pressure
Compressive Strength
Vertical Pressure
(a)
Low Cohesion High Friction
High Cohesion Low Friction
(b)

FIG. 5.—Compression-Tension Diagram and Elements of Compressive Strength.
(a) Compression-tension diagram.
(b) Elements of compressive strength.

The derivation of the Mohr diagram through Coulomb (10),[2] Mohr (10), Prandtl (11), and Buisman (2), and its basic application to bituminous materials by Pfeiffer and associates (10), need not be repeated in detail, but the diagrammatic development is herewith presented for the convenience of investigators (Figs. 3 to 5).

Briefly, a semicircle is inscribed on the horizontal axis, as shown; if only frictional resistance is present, a line tangent to the circle, passing through the origin, has an angle with the horizontal equal to the angle of internal friction, and all such circles are tangent to the same line, which is the "Mohr Envelope." If a constant resistance, independent of loading, is present, the diameter of each circle is increased by a corresponding constant quantity, so that the slope of the line remains constant, but there is now an intercept on the vertical axis; in other words, the origin of the line is shifted. In this case, as shown in Fig. 5(*a*), a negative circle (tensile stress) can be drawn. The full Mohr diagram thus expresses the complete mechanical behavior of the material under the given testing conditions.

Cohesion and Lateral Pressure:

Cohesive material will hold its form without lateral pressure, and the resulting resistance under vertical load is called "compressive strength." It is represented by a Mohr circle where lateral pressure is zero. Noncohesive material cannot do this, but in some cases only a very slight lateral pressure is necessary for high resistance to appear. This is because lateral pressure holds the particles in place; in this sense internal cohesion and external lateral pressure are interchangeable.

The Curved Mohr Envelope:

In practice, the envelope is usually found to be curved. Such curvature makes an analysis impossible because of the number of interpretations possible from a single curved envelope, so long as we assume that only frictional and cohesive resistances are present. This is shown in Fig. 6, (*a*) to (*c*), where it is seen that the curvature could be accounted for by a variation with load, of angle of friction, ϕ, of cohesive shearing resistance, C, or both. If we assume a

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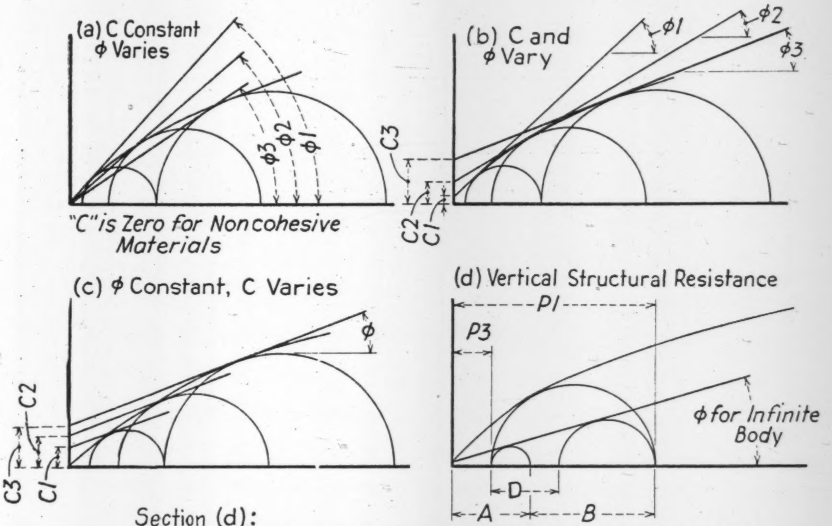
[2] The boldface numbers in parentheses refer to the reports and papers appearing in the Bibliography appended to this paper, see p. 1173.


(a) C Constant
ϕ Varies
$\phi 3$
$\phi 2$
$\phi 1$
"C" is Zero for Noncohesive Materials
(b) C and
ϕ Vary
$\phi 1$
$\phi 2$
$\phi 3$
C3
C2
C1
(c) ϕ Constant, C Varies
ϕ
C2
C3
C1
(d) Vertical Structural Resistance
P1
P3
ϕ for Infinite Body
D
A
B

Section (d):
A = Frictional Part of Vertical Load P1 for Lateral Pressure P3
B = Structural Part of Vertical Load P1 for Lateral Pressure P3
D = Structural Equivalent of Lateral Pressure for P1

FIG. 6.—Interpretations of the Mohr Diagram.

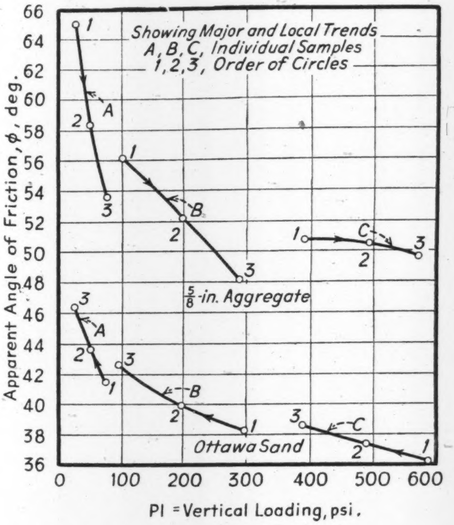


FIG. 7.—Typical Apparent Friction Diagrams.

variation of C, the basic theory is invalidated because we thus assume a variable constant, and a meaning must be given to the diagram other than that assigned it to by Mohr. The simplest approach to the problem is to eliminate the constant altogether by using a noncohesive material, and then investigate the possibility of changes in the angle of friction due to differential loadings.

In shear box tests, it is known that the apparent angle of friction decreases with increased loading. We made tests of the parallel phenomenon as it appears in the triaxial specimen (Fig. 7). The apparent angle of friction is calculated by the method of Fig. 6(a), using the value of C as zero. Here the same tendency appears as is shown in the shear box, but we find the apparent angle of friction tending toward infinity as the load approaches zero. The existence of any true value of ϕ exceeding 45 deg. at the outset appears very dubious. A comparison was then made with some tests made by Hatch (5), who used very large shear boxes (Fig. 8). It will be seen that while the curves are similar, those for the shear box are much flatter, and the apparent values lower. The evidence is very strong for some structural effect which is not true friction and which

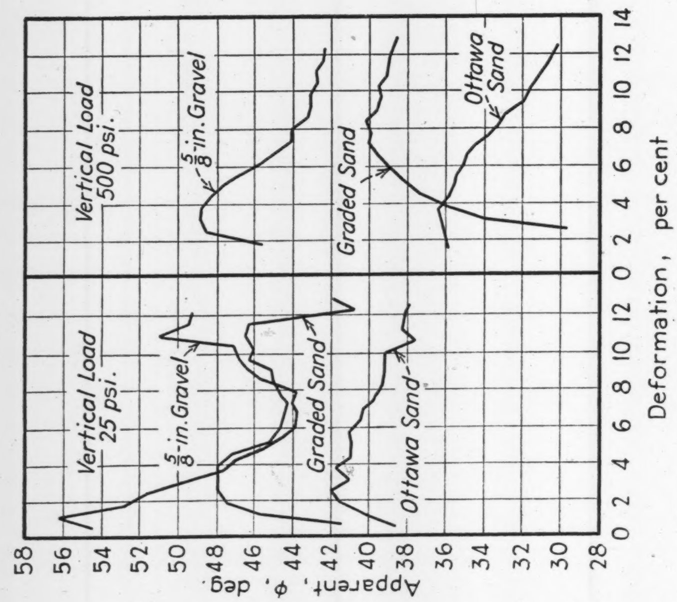


FIG. 9.—Apparent Friction-Deformation Curves.

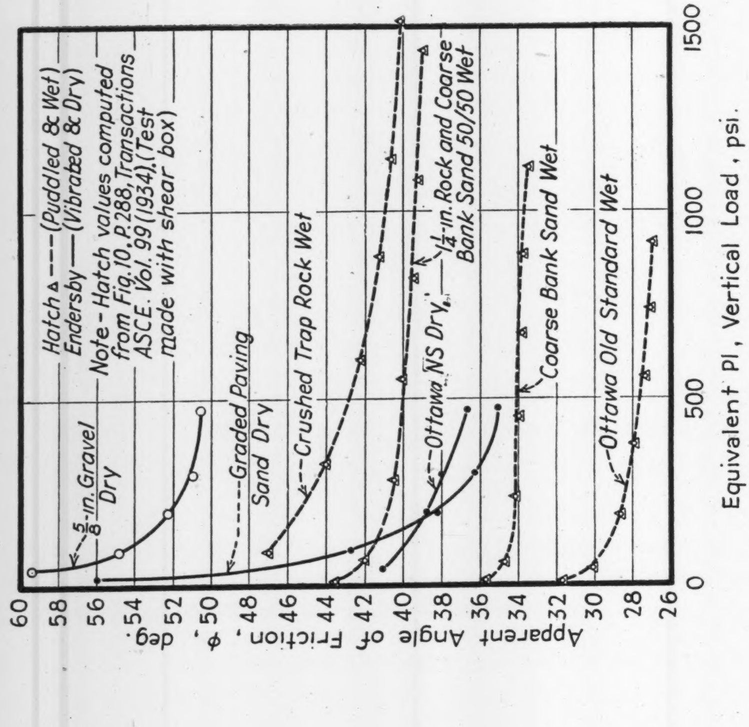


FIG. 8.—Comparative Major Apparent Friction Trends.

is vitally affected by type of apparatus or dimensions or both.

Before investigating these factors, however, it was thought advisable to deal with the theory that the decline of friction with increase of loading in shear boxes is due to polishing or degradation of particles, or both. It was found that such effects were present, at least to some extent. Actual degradation can be observed and measured by sieving. Besides this, when a specimen of normal sand or gravel is used and recompacted several times, the curve for load *versus* ϕ will become somewhat fiatter and lower in position, and the material will become more compactible. Experiments were undertaken for the purpose of discovering the probable magnitude of this effect. It was first reasoned that since extent of deformation, and magnitude of load, must both be factors producing polishing or degradation, variation of one of these factors at a time should be productive of information. If, for instance, loads are decreased while deformation is increasing at a constant rate, and if greatest decrease of friction occurs at highest loads, the curve thus produced should be flatter than that produced by starting with light loads and increasing them as deformation proceeds; and it might even be reversed. Owing to the fact that apparent friction is known to increase as voids decrease, careful study of void changes during deformation was made also. The results of reversing the loads threw little light on the problem of degradation and polishing; it was found that with reversed loading, some flattening of the curve occurred in some—not all—cases; but that the voids were also higher at low loads when loading was reversed, which would presumably produce a similar effect. In no case of compacted materials was the curvature actually reversed or even eliminated. Figure 7 shows, in the case of Ottawa sand, reversed loading; it is typical of numerous other samples so tested.

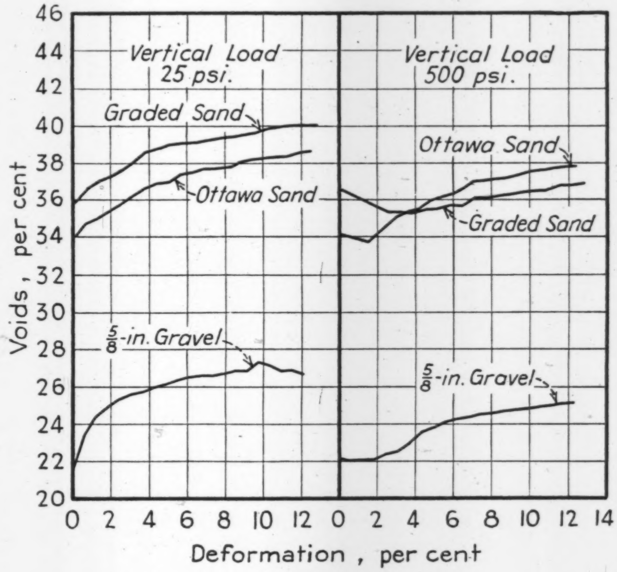


FIG. 10. Void-Deformation Curves.

The effect of prolonged deformation under constant loading was next tried (Fig. 9). It is evident here that something besides friction is present as a resisting factor. There is no systematic decline of apparent friction within the limits of deformation used in previous experiments; the more consistent ultimate decline which is seen would be equally well explained by any other factor which would produce a curved Mohr envelope; and it is hard to imagine that a material which is being polished or degraded under progressive deformation should suddenly become rough and coarse again, which would be the explanation of the sudden reconstitution of resistance seen in several cases. A quasi-rhythmic factor appears in these curves. This also appears in the corresponding void-effect curves (Fig. 10). When a model made of Raschig rings is deformed, it is noted that the rings tend toward a rhythmical increase and decrease of voids as they move out of one formation into another.

All these facts taken together tend to prove a resistance which is not subject to the mathematics of friction, but depends rather upon variable particle arrangements which transmit loads in some other manner. As a final proof, polished bearing balls and lead shot were tested (Figs. 11, 12, 13). These steel bearing balls could not either polish further, or degrade, under the loads used; but they gave an excessively sharp curvature in the Mohr envelope. (One other factor is present: if the specimen is expanding under test, an excess energy input is required at the piston, in order to force the surface outward against the water face. Calculation of this, which is positive in expansion and negative in contraction, showed that it does affect the curvature of the Mohr envelope, but

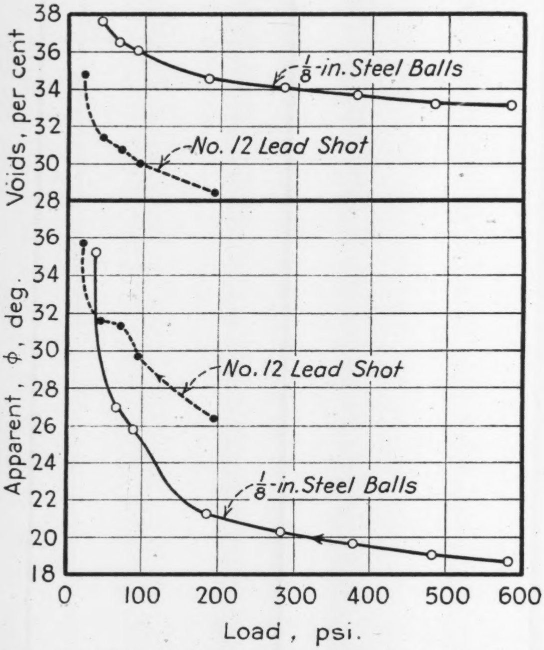


FIG. 11.—Angle of Apparent Friction and Void Content Under Deformation at Decreasing Load

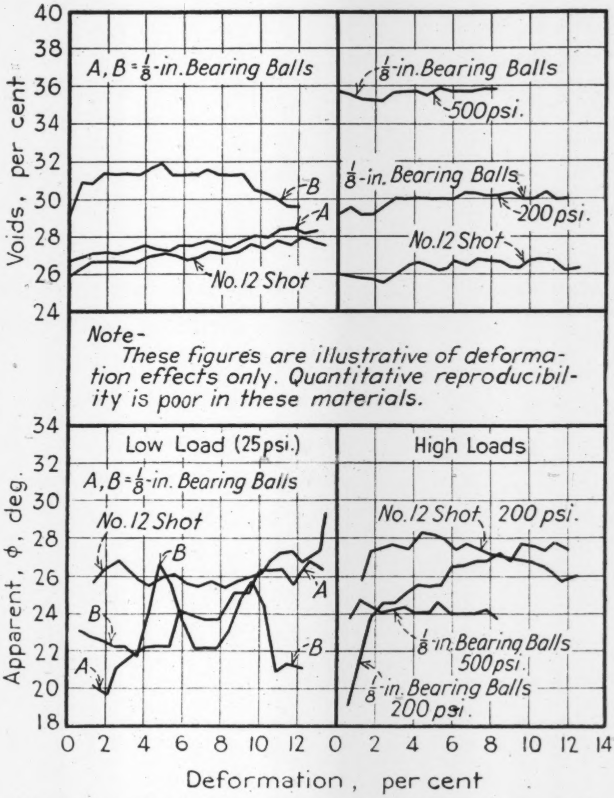


FIG. 12.—Apparent Friction-Deformation and Void-Deformation Curves.

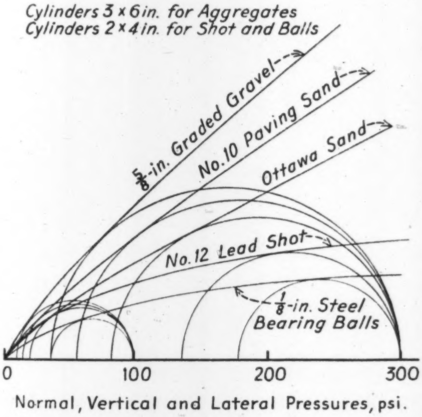


FIG. 13.—Typical Mohr Envelopes for Dry Granular Materials.

NOTE.—These envelopes are averaged from a number of samples under continuous deformation at equilibrium of pressures.

only slightly.) We considered the case proven for the importance of a nonfrictional type of resistance, to which we gave the name "granular structural."

In the literature has appeared recognition of such a resistance; Pfeiffer (10) recognizes an "interlock" as separate from friction and cohesion; Housel (6) has disputed the validity of the idea of "friction" at all, and has advanced a partial mathematical analysis on that basis. In general, "interlock" is often spoken of as one of the forces contributing to friction. Because of that use of the term, we prefer a new one; but because "arch action" (6) appears to be an incomplete designation for the force being discussed, some of the formations being columns rather than arches, we did not use that either.

In order to give a mathematical value to this force, it is now necessary to form a conception of how it acts.

DISTRIBUTION OF PRESSURES IN THE TRIAXIAL TEST

If a bituminized specimen of low height—diameter ratio (H/D) is dissected after deformation, the end cones will be found blunted and the particles on the ends will show stripping and breakage. Thus in specimens of such dimensions we may assume, for the noncohesive as well as the cohesive materials, that the excess resistance over friction is concentrated in the central area. However, we find curved Mohr envelopes for materials where the cones have not interfered according to either visual inspection or theory, that is, where the full section has been sheared without apparent central concentration of resistance. In the case of the low specimens, the blunted, paraboloid cones, with concentrated central stress, are suggestive of true arch action. But, at any dimensions of the specimen, it can logically be assumed that numerous particles will fall into an arrangement whereby lines of stress are transmitted in a zig-zag manner, but with a direct vertical resultant, from the plunger of the apparatus to the base. If the material is moving in full sectional shear, such individual lines would be constantly broken up and reconstituted. In a specimen of considerable height, they would be fairly independent of any reasonable extent of deformation. A simplified representation of the interaction of these factors is shown in Fig. 14, where they are shown in terms of coexistent arch and column action.

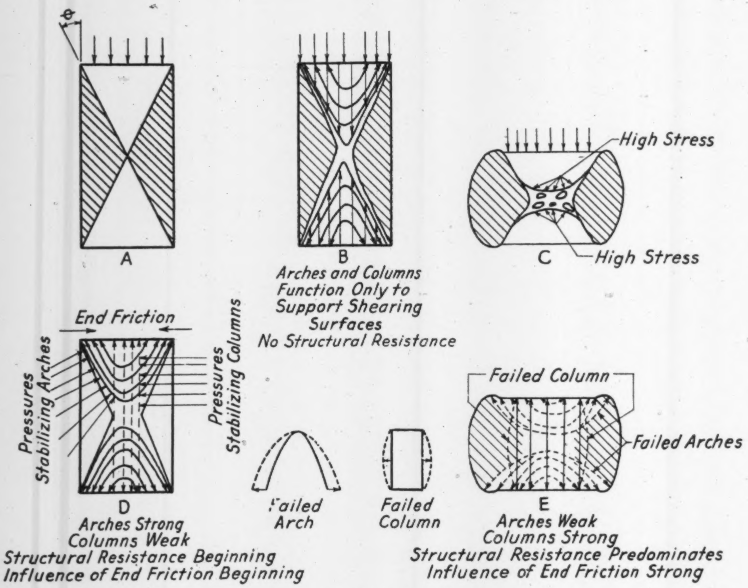

θ
High Stress
A
B
C
High Stress
Arches and Columns
Function Only to
Support Shearing
Surfaces
No Structural Resistance
End Friction
Pressures
Stabilizing Arches
Pressures
Stabilizing Columns
Failed Column
Failed Arches
D
Failed
Arch
Failed
Column
E
Arches Strong
Columns Weak
Structural Resistance Beginning
Influence of End Friction Beginning
Arches Weak
Columns Strong
Structural Resistance Predominates
Influence of End Friction Strong

FIG. 14.—Structural Functions in Cylindrical Test Specimens; Plastic Areas Shaded.

These forces can be considered in the following dimensional stages:

(a) The specimen has a high H/D ratio, exempt from cone interference: In this case arching is inactive, column action is weak and largely independent of extent of deformation.

(*b*) Cones are in moderate interference: Here column action has become rather strong and strong arch action is coming into play. The extent of deformation is beginning to have an effect, as column action is increasing progressively and at the same time the excess stress necessary to break up the original formation of particles in the cones is beginning to contribute to resistance.

(*c*) Cones are in strong interference: Column action has become very strong, arch action is weakening because of the low ratio of height to span, but at the same time increasing force is becoming necessary to rupture the original arrangement of particles in the cones, because the movement of particles is becoming perpendicular to the applied vertical force. Under this condition also, the arch resistance is increasingly dependent upon the friction between the material and the base, and upon the presence of cohesion in a cohesive material. In some cases the rapid increase of column strength, and the force necessary to rupture increasing cross-section of the cone, produces a constantly rising stress-strain curve without any maximum. This is the opposite of the condition in a tall specimen, where the stress-strain curve tends toward a straight line independent of strain.

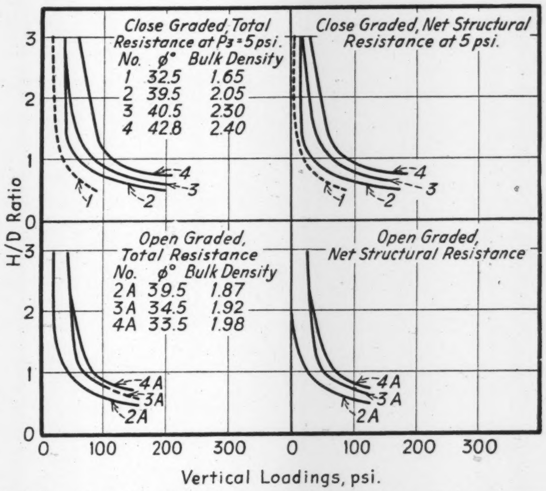


FIG. 15.—Structural Influences in 4-in. Diameter Specimens.

MOHR ANALYSIS OF GRANULAR-STRUCTURAL RESISTANCE

The case would be hopelessly complex if it were necessary to completely separate all the above factors in resistance. For practical purposes it is only necessary to recognize that their resultant is vertical and to assume that they are primarily dimensional. A series of Mohr envelopes at changing dimensions will then produce an extrapolation curve giving an approximately true value of angle of friction (approaching the value in an infinite body). We can then use this value to draw a basic straight envelope passing through the origin, and this leads to the solution shown in Fig. 6 (*d*).

Examples of the Application of the Analysis:

Four dry aggregates, each (with the exception of No. 1) graded to the same type of maximum density curve and so constituted that each formed a fraction of the next largest size, were synthesized to maximum screen sizes of No. 200, No. 10, ½ in., and 1 in. In turn, particles passing No. 50 screen were removed from the No. 10 grading, producing aggregate 2A, and particles passing No. 10 screen were removed from each of the larger gradings, producing aggregates 3A and 4A. These were then tested in the 4-in. diameter apparatus at various H/D ratios. The results in total and structural stabilities are shown in Fig. 15. In only one case, of the open-graded sand, was a purely frictional resistance (straight envelope) found, though the No. 200 dust almost reached that status.

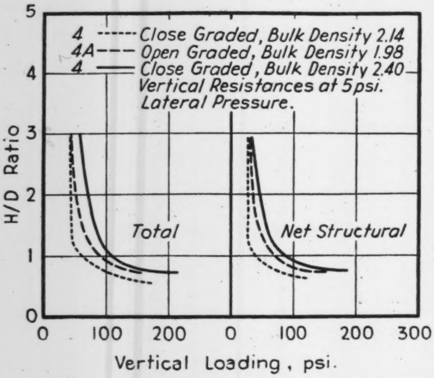


FIG. 16.—Influence of Bulk Density Variations on 1-in. Aggregate.

The following points were noteworthy:

Angles of Friction.—All three close-graded aggregates have angles of friction differing little within the limits of accuracy of such methods. The open-graded sand has the same angle as the close-graded sand. The two open-graded gravels have lower angle of friction than the close-graded aggregates, but resemble each other. In the close-graded materials, friction increases slightly with large increase of bulk density, while in the open-graded it decreases greatly with a light *increase* of bulk density. This leads to two suppositions:

(*a*) That density affects structure greatly but affects friction little;

(*b*) The gravel fractions have less inherent friction than the sand. The exact contribution of the fines to the friction of the mix is dubious. They may act in somewhat the same manner as grit in a bearing, presenting resistance to movement by the force necessary to roll angular particles between larger surfaces.

Comparison of the shapes of envelopes for the close and open-graded mixes also indicates that loading in the close-graded material may not have been carried out far enough to reach a true value of friction (Fig. 19).

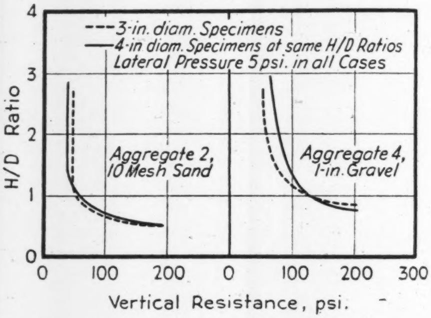


FIG. 17.—Influence of Variation of Specimen Diameter.

Maximum Particle Size.—The influence of these differences is slight at great H/D ratios, great in low specimens whose dimensions correspond with road practice.

Types of Density.—Density may be varied by changing the grading, by changing the degree of compaction, or by deforming under triaxial pressures. Typical results of variations for aggregate No. 4 are shown in Fig. 16. Lack of compaction acts like reducing the maximum particle size; and the convergence of structural resistance of the different densities around $H/D=3$ is noticeable. The influence of changes of density during deformation cannot be described easily.

Influence of Absolute Dimensions of Specimens.—Our investigation of the influence of absolute dimensions of specimens has been brief; initial results as shown in Fig. 17 should be regarded with caution. If further substantiated, the peculiar relationship between the curves for the 3-in. and for the 4-in. diameter cells may be ascribable to the complex relations between slenderness ratios of individual columns within the material and the mass column formed by the whole specimen, also the variable influence of arch and column action. Figure 17 is introduced at this stage to call attention to the fact that results on the same material with equipment of different dimensions may be far apart. In all such cases the smaller specimen is less likely to be representative.

The foregoing results are important in both soil mechanics and bituminous work; they indicate that results between investigators are not comparable unless methods are standardized, that results of present testing methods must be correlated on a dimensional basis for correct application to field conditions, and particularly that any method of testing bituminous paving materials without correlating dimensional effects with road conditions is far afield.

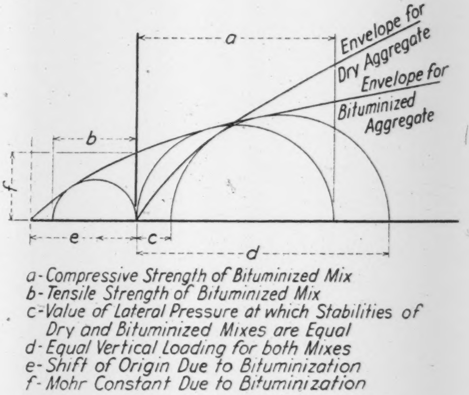
The fact that in many mechanical tests we are examining the properties of a particular object (the specimen), as well as those of the material of which it is composed, is especially exemplified by such results.

THE EFFECT OF ASPHALT

Bitumen is not simply a cohesive whose true effects in a mix can be measured by such methods as the penetrometer. For analytical information on this, reference is made to the companion paper by T. F. Ford (**4**).

In general, asphalt produces two opposite effects on stability, reducing it in so far as it acts as a lubricant and increasing it in so far as it acts as a cohesive. Complex effects result from this paradox. Furthermore, speed and temperature effects must be taken into consideration.

In the Mohr diagram, this dual behavior of bitumen results in a shift of the axial origin and sometimes causes the envelopes of the bituminized and that of the dry mineral aggregate to cross at a lateral pressure where the stabilities of the two may be equal under a certain testing speed or temperature. There is a straighter envelope with the bituminized material, because the shift of axis throws the positive position of the circles farther out on the normally straighter part of the envelope (Fig. 18).


a
b
c
d
e
f
Envelope for Dry Aggregate
Envelope for Bituminized Aggregate

a - Compressive Strength of Bituminized Mix
b - Tensile Strength of Bituminized Mix
c - Value of Lateral Pressure at which Stabilities of Dry and Bituminized Mixes are Equal
d - Equal Vertical Loading for both Mixes
e - Shift of Origin Due to Bituminization
f - Mohr Constant Due to Bituminization

FIG. 18.—Relation of Mohr Envelopes for Dry and Bituminized Mixes.

To illustrate these effects, aggregates Nos. 3 and *3A* are shown dry and bituminized (Figs. 19 and 20):

1. *Composition.*—These aggregates show probably the extremes of grading effects in $\frac{1}{2}$-in. gravels and are not presented as road materials. The asphalt used for illustration was an 85/100 Cali-

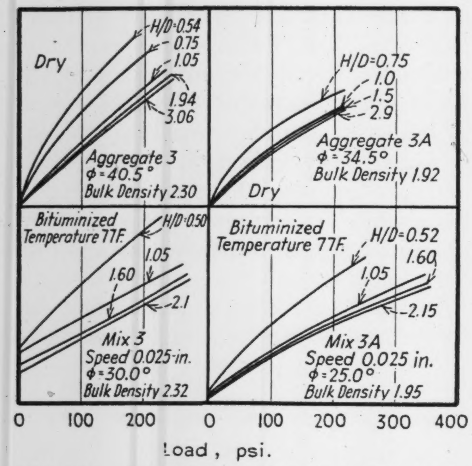


FIG. 19.—Typical Mohr Envelopes; Dry and Bituminized Materials; Temperature 77 F.

fornia of Newtonian type, which presents the simplest stability phenomena. Four parts were added to 100 parts aggregate in each case, by weight.

2. *Fabrication.*—Dry aggregates were vibrated to constant density in a high-frequency oscillator. Mixes were compressed at 1500 psi., in 4-in. diameter cylinders, in separate segments 2 in. high, temperature 120 F., and pressure held for 1 min. The material was vibrated during initial application of pressure.

3. *Density Phenomena.*—Both mixes lost structural or aggregate density by bituminization, the open-graded mix least. (This effect appears typical and is logical.) In both mixes as well as in the aggregates, density decreased upon initial deformation and thereafter increased. The effect of increase is to straighten the Mohr envelope somewhat, more so in the case of the mix than the aggregate. In one case, as will be noted, this effect caused a reversal of curvature. Given constant lateral pressure, densities become or remain higher as the height of specimen decreases.

4. *Friction.*—The effect of increasing density during deformation being to straighten the envelope, true friction would be somewhat less than the observed angles under the method used. Friction loss in mix 3 was 10.5 deg. and in mix 3*A* was 9.5 deg.

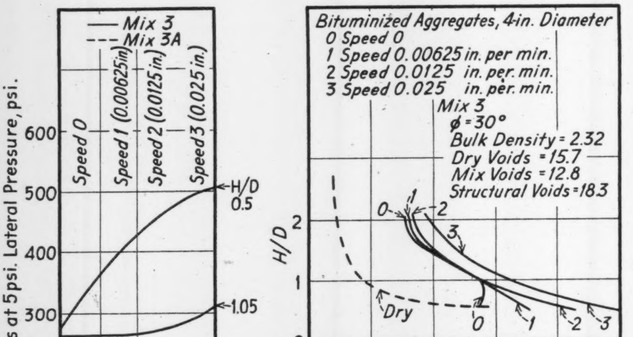


FIG. 20.—Speed-H/D Effects.

5. *Viscosity, Speed, Temperature, and Cohesion.*—Lowering the speed of shear in the mix parallels lowering the lateral pressure in the dry aggregate, because viscous resistance decreases with speed. It also parallels the result of increased temperature. But as is seen in the Mohr envelopes, lowering the lateral pressure results in increasing the *proportion* of structural resistance. Hence, in the greater heights of specimen, the characteristically curved extrapolation line results; but this curvature is also influenced by capillary and other effects which leave a residual cohesion at zero speed; otherwise the static stability of a mix would always be lower than that of the aggregate. The open mix shows far less cohesion and apparent cohesion because of lack of contact points and lack of capillary effects, though its entire set of relationships resembles a scaled-down repetition of those of the close-graded mix.

In considering the effect of speed, the lower height specimens develop shear forms which result in faster virtual shear of the material at the same testing speed. This causes an additional viscous effect at these low heights, increasing stability. The spread between speed zero and the other speeds at $H/D = 0.5$ appears due to the fact that all virtual speeds here are so much greater than the measured speeds, while of course zero measured speed and zero virtual speed are the same.

6. *Height to Diameter Ratio* (H/D) *Effects.*—In the case of mix 3, the difference between the curve for dry materials and the curves for bituminized materials at high testing speeds (Nos. 2 and 3) is the type of difference which results from increasing the lateral pressure. In the curves for bituminized materials at lower speeds, arch failure at low heights sets in and becomes extreme for the zero speed. (These phenomena are repeated for mix 3*A* in a less marked manner.) The apparent reason for the arch failure is as follows: at a given H/D ratio, the ratio of rise to span of the arches in the unsheared cones becomes so low that the arches become quite unstable. This point depends, among other things, on the coefficient of friction between the material and the loading surface. (Pfeiffer (10) shows the weakening of a low specimen by the interposition of rubber on the contact surface.) In addition to the contact friction and the lateral pressure, the cohesion of the mix buttresses these arches; but at zero speed this cohesion becomes very low. Hence this combination of influences produces in mix 3 an *S*-curve for zero speed, and in mix 3*A* a tendency in the same direction. That the curve does not reverse in the latter case appears to be due to the very low viscous resistance of the mix to begin with. It should not be assumed that the effect in a pavement would be the same; probably contact friction would be different. Figure 24 shows an arch failure under the plunger in a bearing test. With a slight variation of contact friction, we find the arch failure on the subgrade instead.

7. *The "Macadam Point."*—The stability curve for the dry material intersects all curves for bituminized material at certain heights of sample, where the asphalt no longer acts as a stabilizer; here the "macadam principle" predominates, asphalt being merely a protecting agent at lower heights. This need not be the corresponding thickness of pavement, although there is a correlation.

8. *Working Values.*—Mix 3 is an aggregate of great stability such as would seldom be encountered in practice and is too sensitive for road use. Mix 3*A* is of a type rather resembling an extreme practice which is reported to be in some use in the West. These extremes were selected to illustrate the principles.

Ottawa sand represents the ultimate in open grading and low surface friction, and like some sands with which disastrous experience has been encountered, cannot be made statically stable with any ordinary asphalt; asphalt only lubricates it.

USE OF THE STRESS-STRAIN DIAGRAM

Stress-strain diagrams resulting from the triaxial deformation of samples under constant lateral pressure are used in soil mechanics. In both dry and bituminized aggregates they give a typical curve, subject to dimensional effects, illustrated by Fig. 21. The form of this is so much like that of an elastic-plastic material that it can be easily taken for such in bituminized materials. True elastic phenomena appear, however, only under very high speeds or low temperatures. The rise of the curve, as mentioned, is evidently due to the fact that the particles are pushed into more stable formations as deformation proceeds, but it is curious that such a rise is usually accompanied by increasing voids. This could best be explained by a "toggle-joint" action. As the Mohr envelope straightens with increasing H/D ratios, the initial rise of the stress-strain diagram tends to vanish. This form of diagram can be used analytically to plot Mohr diagrams at constant extent of deformation, constant void content, etc.

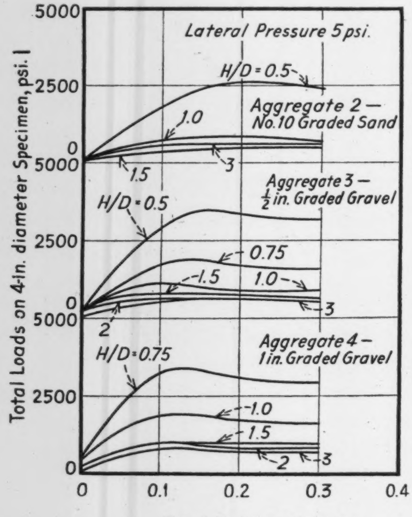


FIG. 21.—Stress-Strain Curves for Various H/D Ratios.

Four stability factors appear in stress-strain diagrams: initial slope of curve, yield point, maximum load, ultimate load—the three latter to be considered in connection with extent of deformation. The relative weighting to be given these has yet to be established. Since the area under the curve represents the energy required to produce a given deformation, this factor may be productive of practical correlations.

STABILITY UNDER SUCCESSIVE LOADINGS

The stress-strain diagram, plus the time element which governs the viscous effect of the asphalt, permits an approach to the problem of road stability. Apparently five conditions may theoretically exist:

1. The structure of the aggregate itself can carry the load with only elastic deformation. In this case bitumen is needed only for protection, not cohesion, and initial stability is complete.

2. The structure can carry the load after a given deformation. Upon application of load, it deforms until such a deformation is reached, and then becomes stable; bitumen delays that deformation.

3. The resistance of the binder is high so that, in combination with the resistance of the structure, it permits only elastic deformations, and initial stability is again complete.

4. The structure alone will not carry the load at any deformation, but a very hard binder is present which so delays

the failure that apparent stability is present for a long time.

5. The material is not truly stable, but owing to the cross-traverse of traffic and continuous reformation and reconsolidation, instability is not visible. The principal effect of this condition is degradation of the aggregate.

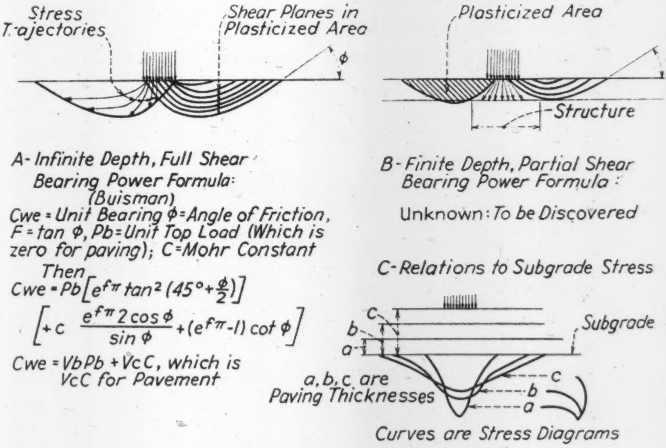

Stress Trajectories
Shear Planes in Plasticized Area
ϕ
Plasticized Area
ϕ
Structure
A- Infinite Depth, Full Shear Bearing Power Formula: (Buisman)
Cwe = Unit Bearing ϕ = Angle of Friction, F = tan ϕ, Pb = Unit Top Load (Which is zero for paving); C = Mohr Constant
Then
$Cwe = Pb\left[e^{f\pi}\tan^2(45^\circ+\frac{\phi}{2})\right]$
$\left[+c\ \frac{e^{f\pi}2\cos\phi}{\sin\phi}+(e^{f\pi}-1)\cot\phi\right]$
Cwe = VbPb + VcC, which is VcC for Pavement
B- Finite Depth, Partial Shear Bearing Power Formula:
Unknown: To be Discovered
C- Relations to Subgrade Stress
c
b
a
Subgrade
a, b, c are Paving Thicknesses
c
b
a
Curves are Stress Diagrams

FIG. 22.—Prandtl's Theory for Strip Bearing.

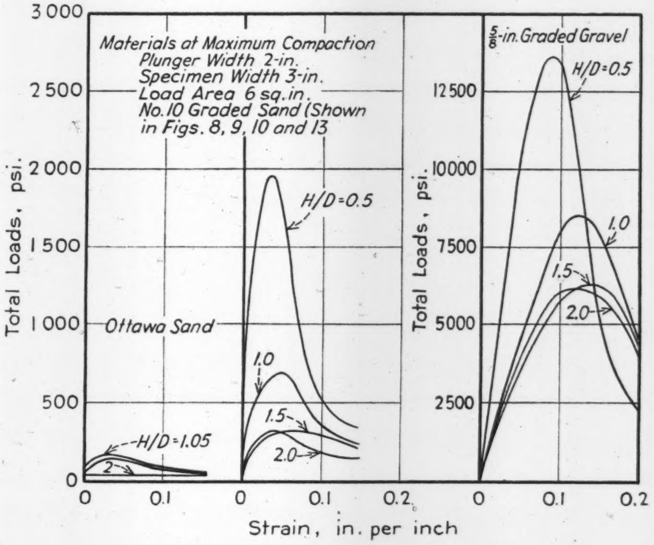


FIG. 23.—Strip Bearing Load Stress-Strain Curves.

APPLICATION OF THE PRINCIPLES TO OPEN SURFACES

The formulas developed by Prandtl (10, 11) and Buisman (2) for bearing on open bodies of unlimited depth have received substantial verification. These formulas are insufficient where structural resistance is present. The respective relationships are shown in Fig. 22, where case *B* involves a rigid base. A third case, not shown, is that of compound Prandtl shear through both base and pavement.

We have taken some stress-strain curves in bearing tests, which are shown in Fig. 23, with the method illustrated by Fig. 24. In this test noncohesive materials tend to develop a distinct shear plane, while bituminized materials do not. It will be noted that increasing thinness results in increasing sharpness of yield point, that is, brittleness.

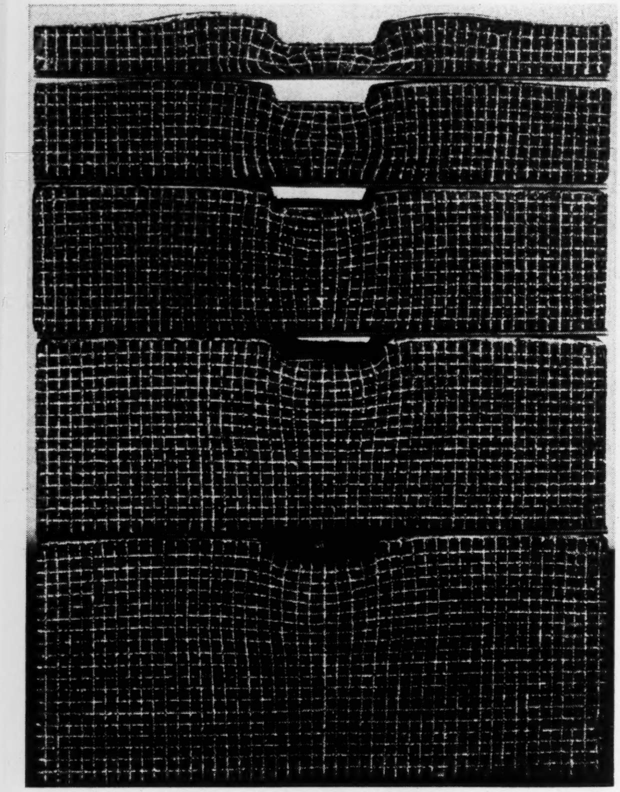


FIG. 24.—Deformation Phenomena Under Bearing Test.

CONCLUSIONS

1. For correct analysis of the stabilities of granular and granular-plastic materials, it is necessary to recognize, and measure separately from friction and cohesion, a type of structural resistance which is strongly dependent upon the dimensions of the specimen.

2. It is necessary to measure the stability of the entire mix, not of fractions.

3. It is necessary to measure stability under dimensional conditions correctly related to actual loading conditions.

4. It is impossible to measure completely or to predict the behavior of bituminous road materials by any test which is single-point as to dimensions, temperature, or speed.

5. The most suitable method of investigation is the triaxial test with Mohr

circle analysis, supplemented by some form of bearing tests.

6. The bearing power of pavement mixes cannot be correctly evaluated without taking into consideration the influence of the contact plane between pavement and subgrade.

7. It is apparent from the literature that considerable study has been given to the effect of pavement thickness upon the subgrade load, but inadequate study to its effect upon the internal stability of the pavement itself. None of the current formulas was found applicable to our test results except under special conditions.

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THE MEASUREMENT OF THE DEFORMATION PROPERTIES OF ASPHALTS

By T. F. Ford[1] and K. G. Arabian[1]

Synopsis

The characterization of the deformation behavior of asphalts by the penetration test or by other empirical tests is recognized as being inadequate, particularly for asphalts which are elastic as well as viscous. The rotating cylinder type of viscosimeter permits the separation of elastic and viscous effects, since it permits observation of changes in deformation rate with deformation itself. Other methods are in general open to the objection that the sample is subjected to complex deformations. Rotating cylinder viscosimeters, however, are usually considered too time-consuming for the results obtained. Here a simple instrument of this type is reported, which is designed for routine use. Results obtained using this instrument compare favorably with results obtained using a more elaborate conicylindrical viscosimeter. Since a single deformation constant such as viscosity does not completely describe the flow of plastic asphalts, three deformation constants are evaluated: a coefficient of viscosity *n*, a coefficient of elasticity in shear *g*, and a coefficient of internal slip *s*.

Asphalts are universally characterized by the penetration and softening point tests (Methods D 5 - 25 and D 36 - 26).[2] Other empirical tests such as the ductility test (Method D 113 - 39)[3] also are widely used. None of these are fundamental in nature nor adapted to direct correlations one with the other.

The inadequacy of the commonly used tests as measures of consistency is recognized (**1, 2, 3, 4, 5, 6, 7, 8, 9, 10**).[4] Asphalts of equal penetration may differ many fold in apparent viscosity. Whereas some are purely viscous, others are highly elastic so that under constant loads deformation rate changes with deformation itself. Thus equations for the calculation of viscosity from the penetration (**5, 8**) are not applicable to nonviscous asphalts. The penetration test measures an over-all deformation rate, and in the absence of other tests it does not disclose effects of deformation itself. Classifications of asphalts according to relationships between penetration and softening point (**9**) recognize this fact and in effect characterize asphalts as to relative deviations from viscous behavior. Equations for calculating viscosities from penetration, and arbitrary classifications based on relationships between empirical tests, such as between penetration and softening point, have been developed because these empirical tests are simple and rapid. The direct measurement of the absolute flow properties of asphalts has been handicapped by the lack of simple absolute testing methods.

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[1] Member of Research Staff, Shell Development Co., Emeryville, Calif.

[2] Standard Method of Test for Penetration of Bituminous Materials (D 5 - 25), 1939 Book of A.S.T.M. Standards, Part II, p. 412; Part III, 185.
Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 - 26), 1939 Book of A.S.T.M. Standards, Part II, p. 417.

[3] Standard Method of Test for Ductility of Bituminous Materials (D 113 - 39), 1939 Book of A.S.T.M. Standards, Part II, p. 398.

[4] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1181.

Various Instruments:

Many methods for measuring flow properties in absolute units have been proposed. A review describing a number of these has been given by Saal (5). To all of them except the rotating cylinder viscosimeter there are objections which arise primarily from the fact that for nonviscous asphalts deformation behavior varies with deformation itself. In the Vogel-Ossag capillary method or in pressure capillary methods (3, 11), and in falling ball (6) or sinker methods the nature of the deformation varies throughout the sample. In the Pochettino falling cylinder (12), or the Bingham-Stephens alternating stress viscosimeter (2), or by the method of deforming rods, the sample is constantly altered in cross-section. By all of these methods the extent of deformation in one direction is limited. It is only in the rotating cylinder type of instrument that the shearing stress may be held constant while the properties are determined with progressive deformation in one direction.

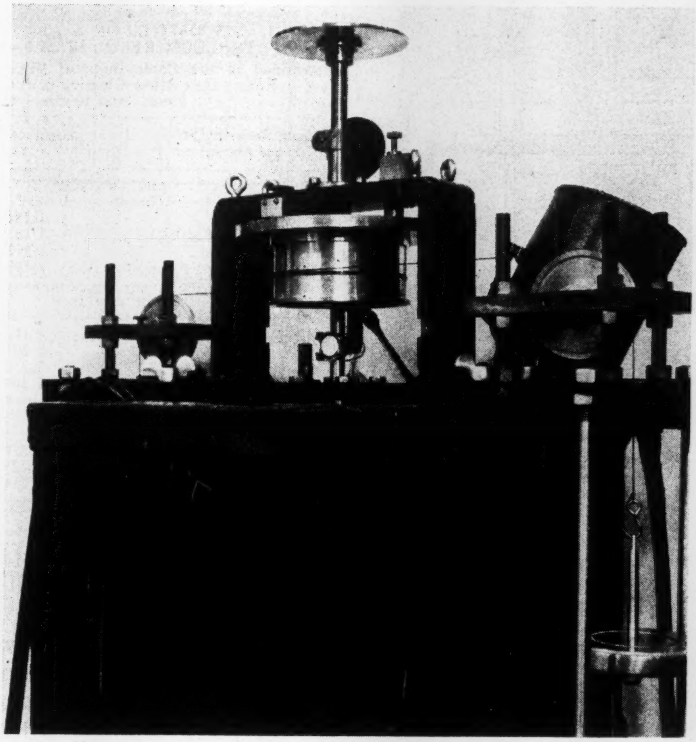


FIG. 1.—Assembly of Conicylindrical Viscosimeter.

Rotating cylinder or Couette type viscosimeters have been widely used for research studies of the flow properties of asphalts (3, 5, 8, 10). Troublesome end effects can be eliminated mathematically by using cylinders with conical bases as proposed by Mooney and Ewart (13).

Lee, Warren, and Waters recently reported a viscosimeter of this type (10). Our own conicylindrical viscosimeter, which is quite similar, is shown by Figs. 1 and 2.

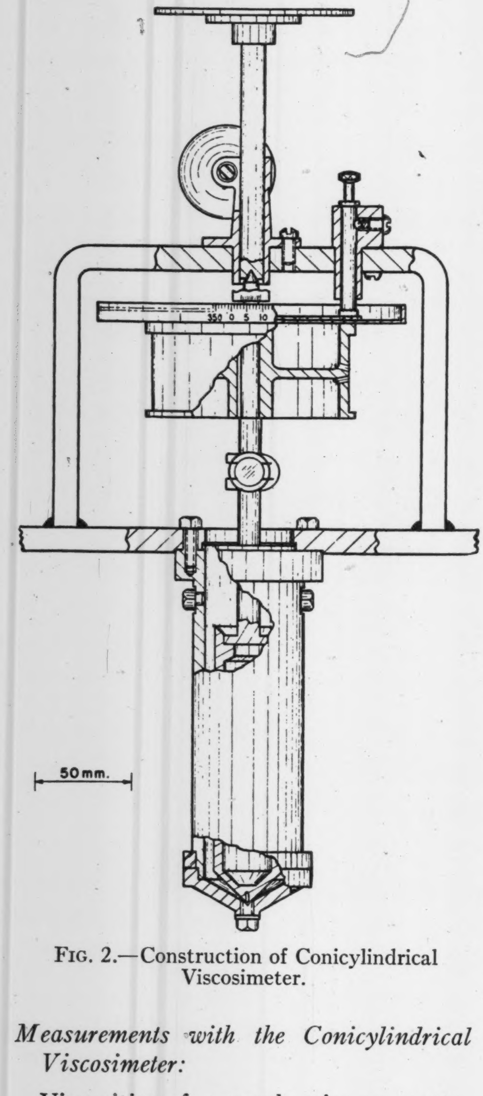


FIG. 2.—Construction of Conicylindrical Viscosimeter.

Measurements with the Conicylindrical Viscosimeter:

Viscosities of a purely viscous petroleum resin, as measured in our conicylindrical instrument, are given in Table I. For shearing stresses varying seven-fold the average deviation from the mean viscosity is 0.16 per cent using a rotor giving 1 mm. clearance between cylinders, 0.35 per cent for a 5-mm. clearance, and 0.11 per cent for a 10-mm. clearance. The small increase in apparent viscosity with increasing clearance may be attributed to the effects of the corners at the junction of the conical and cylindrical surfaces. These data for a purely viscous material show the high degree of accuracy possible with this type of instrument.

TABLE I.—VISCOSITIES OF A 146 PENETRATION PETROLEUM RESIN AT 25 ± 0.02 C.

Determined in the Conicylindrical Viscosimeter Using Rotors that Allow Clearances of 1 mm., 5 mm., and 10 mm.

Mean Shearing Stress, dynes per sq. cm.	Absolute Viscosity, poises
1 mm. clearance	
3 950	3.184×10^5
8 470	3.185×10^5
16 100	3.172×10^5
27 300	3.188×10^5
5 mm. clearance	
4 920	3.177×10^5
10 550	3.201×10^5
20 100	3.212×10^5
34 810	3.209×10^5
10 mm. clearance	
8 468	3.217×10^5
18 100	3.221×10^5
34 400	3.211×10^5

The curves of Figs. 3 and 4, also obtained with this instrument, illustrate extreme deviations from viscous flow as exhibited by a Mexican blown asphalt. The rapidly decreasing deformation rate with deformation, as well as the marked recovery on release of the driving loads, indicate elastic forces. These curves show that for such materials the deformation over an arbitrarily specified time, as obtained by the penetration test, has no direct relation to viscosity. They also show that values obtained in capillary viscosimeters, which are a complex measure of initial and equilibrium rates of flow, are inadequate as gages of deformation behavior. Although such truly descriptive measurements as illustrated by Figs. 3 and 4 can be obtained with the conicylindrical viscosimeter, the complexity of this instrument and the time required for measurements make it impractical for routine use.

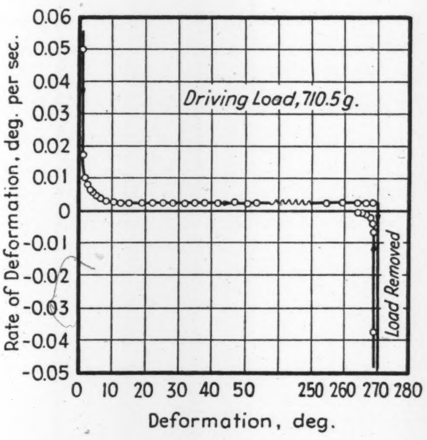


FIG. 3.—Conicylindrical Viscosimeter Measurements of Deformation and Recovery of a Mexican Blown Asphalt; Tests at 250 C. ± 0.2 C., and 10,400 dynes per square centimeter Shearing Stress.

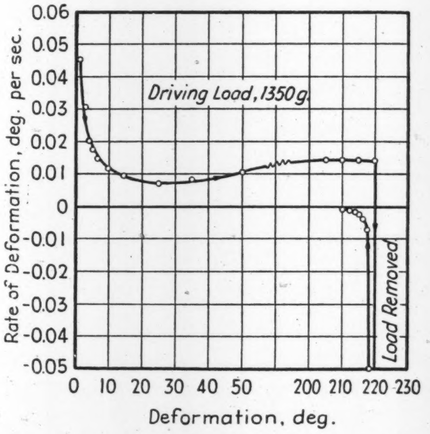


FIG. 4.—Conicylindrical Viscosimeter Measurements of Deformation and Recovery of a Mexican Blown Asphalt; Tests at 25 C. ± 0.2 C., and 19,880 dynes per square centimeter Shearing Stress.

A Simple Rotating Cylinder Viscosimeter:

The instrument shown in Figs. 5 and 6 retains the theoretical advantages of the rotating cylinder design. Essentially

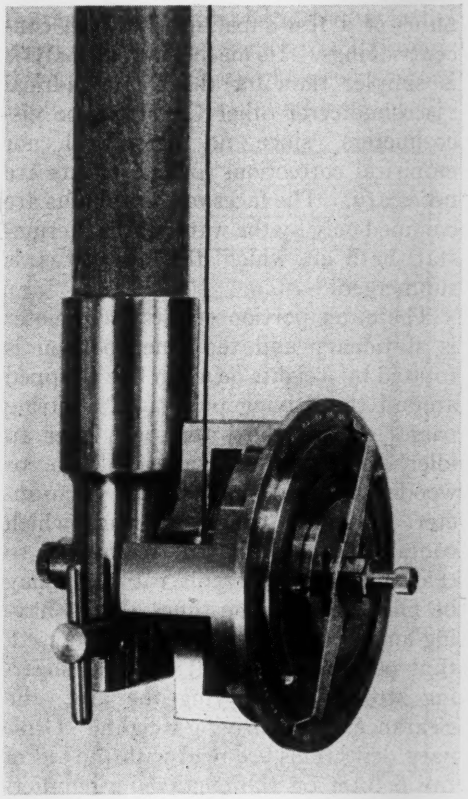
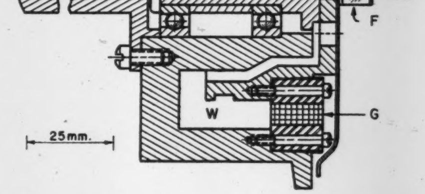


FIG. 5.—Assembly of Open-End Viscosimeter.



A	Stationary frame.	F	Hexagonal mirror.
B	Pointer.	G	Asphalt sample
C	Stationary removable ring.	H	Pulley.
D	Rotating removable ring.	Q	Oil.
E	Rotor.	W	Water.

FIG. 6.—Construction of Open-End Viscosimeter.

it provides a means for twisting an annulus of a test substance between concentric rings. Its mathematical analysis is simpler than for the conicylindrical viscosimeter or other Couette type viscosimeters, since no theoretical nor empirical corrections for end effects are necessary. The faces of the annulus are confined only by the water of the thermostat bath in which the apparatus is submerged.

The outer portion of the viscosimeter is stationary and the inner portion is rotated by weights on a fish line wrapped around the driving pulley. The string passes outside the water bath over an idler pulley on the same bakelite or wooden rod that supports the viscosimeter. The two concentric rings which contain the sample are removable so that any number of replaceable sets may be filled at the same time. Rings having any desired clearances may be used, thus permitting tests at different shearing stresses by varying the area, the clearance, or the driving weights. Ordinary deflections are read with the aid of the pointer on the engraved circumference of the instrument. An optical system may be provided for the magnification of small deflections.

The rotor turns on steel ball bearings in light oil which fills the bearing housing. The bearing oil like the sample tested is retained in the housing by hydrostatic pressure. With care in removing the viscosimeter from the water bath the oil will not be lost. Moreover it will not become contaminated with water unless the rotor is turned at excessive speeds.

The asphalt is introduced molten between the removable rings, using suitable filling blocks to hold the rings concentric while the sample cools. The upper surface of the cold asphalt sample may be trimmed flat with a hot knife. The lower surface may be prevented from sticking to the filling block by using a mixture of talc, glycerine, and liquid soap. Since the rings are tapered for easy insertion in the instrument they must be filled with the narrow sides up. The screw holes must also be aligned before filling. With hard asphalts the filled rings are easily inserted in the instrument, but even with liquid asphalts the operation is not difficult if conducted under water.

Data are given in Table II comparing viscosities of purely viscous materials (ranging from 12 to 257 penetration) as measured in this simple viscosimeter and in the conicylindrical viscosimeter; agreement in these and other cases is within 1 to 4 per cent. The material of 257 penetration is a heavy road oil.

TABLE II.—VISCOSITIES OBTAINED IN THE CONICYLINDRICAL VISCOSIMETER AND IN THE OPEN END VISCOSIMETER USING PURELY VISCOUS MATERIALS AT 25 ± 0.02 C.

Determined in the Conicylindrical Viscosimeter		Determined in the Open End Viscosimeter	
Mean Shearing Stress, dynes per sq. cm.	Absolute Viscosity, poises	Mean Shearing Stress, dynes per sq. cm.	Absolute Viscosity, poises
12 Penetration Petroleum Resin			
10 400	5.30×10^7	15 250	5.27×10^7
19 880	5.34×10^7	30 700	5.24×10^7
34 600	5.28×10^7	38 800	5.42×10^7
86 Penetration Petroleum Resin			
4 860	8.35×10^5	3 760	8.39×10^5
19 880	8.39×10^5	15 400	8.36×10^5
34 600	8.35×10^5	38 800	8.32×10^5
146 Penetration Petroleum Resin			
10 400	3.20×10^5	15 250	3.37×10^5
19 880	3.21×10^5	30 700	3.36×10^5
34 600	3.21×10^5	38 800	3.33×10^5
257 Penetration Road Oil (SC-6)			
10 400	9.02×10^4	15 250	9.26×10^4
19 880	9.04×10^4	30 700	9.40×10^4
34 600	9.02×10^4	38 800	9.25×10^4

The curves of Figs. 7 and 8, obtained with this simple instrument are for the same Mexican blown asphalt represented by the curves of Figs. 3 and 4 obtained with the conicylindrical viscosimeter.

Similar comparisons for other nonviscous asphalts have been obtained.

The Evaluation of Deformation Constants from Rotating Cylinder Viscosimeter Data:

With concentric cylinder viscosimeters, the viscosity of purely viscous materials may be calculated from the speeds of rotation at given loads, and the elasticity of purely elastic materials may be calculated from stress-strain curves.

The single proportionalities represented by the coefficients of viscosity or elasticity do not describe complex plastic deformations such as those illustrated by the curves of Figs. 3, 4, 7, or 8. A minimum characterization of such curves requires a coefficient n, determined by the initial speed of deformation or recovery, a coefficient g determined by the initial slope of the deformation or recovery curve, and a coefficient s determined by the equilibrium speed of deformation.

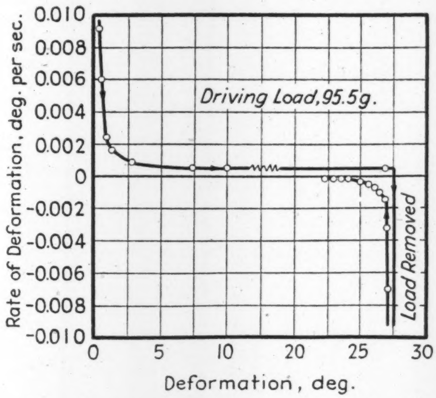


FIG. 7.—Open-End Viscosimeter Measurements of Deformation and Recovery of a Mexican Blown Asphalt; Tests at 25 C. ± 0.2 C., and 3760 dynes per square centimeter Shear Stress.

Behaviors such as illustrated by the curves of Figs. 3, 4, 7, and 8 suggest a model consisting of loosely connected elastic elements embedded in a viscous medium, which are extended during deformation and may slip relative to one another. In such a model, the initial speed and thus the coefficient n is proportional to the viscosity of the continuous liquid medium; the initial rate of decrease in speed, and thus the coefficient g, is proportional to the elasticity of the elastic elements initially extended; and the equilibrium speed or rate of slip,

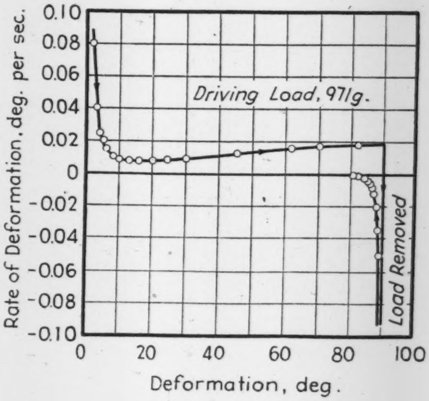


FIG. 8.—Open-End Viscosimeter Measurements of Deformation and Recovery of a Mexican Blown Asphalt; Tests at 25 C. ± 0.2 C., and 38,300 dynes per square centimeter Shearing Stress.

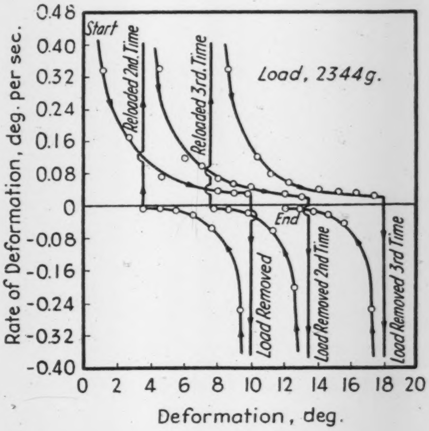


FIG. 9.—Reproducible Elastic Behavior of Mexican Blown Asphalt; Tests at 25 C. ± 0.02 C., and 35,000 dynes per square centimeter Shearing Stress.

designated by the coefficient s, is proportional to the sum of all the resistances to continued deformation when the elastic structure has been fully extended.

The analysis suggested is similar to one developed by Poole (**14**), except that it also includes the possibility of slip, which is necessary when dealing with asphalts. This is because recovery occurs even after extended deformations; because, in general, recovery follows the same pattern as the deformation; and because successive deformation and recovery curves may be reproducible but displaced from the original axis, as shown in Fig. 9. Under high loads the speeds may increase somewhat after reaching a low value, as illustrated by the curves of Figs. 4 and 8; this may be attributed to partial failure of elastic structures.

TABLE III.—DEFORMATION CONSTANTS OF VARIOUS ASPHALTS AND OTHER MATERIALS, CALCULATED FROM OPEN END VISCOSIMETER DATA.
(Temperatures 77 ± 0.04 F.)

Material Tested[a]	Load, g.	n, poises[b]	s, poises[b]	g, dynes per sq. cm. [b]
Mexican steam reduced asphalt:				
Penetration, 103	971	4.0×10^5	11×10^5	12×10^4
Softening point, 119.5	95.5	6.0×10^5	13×10^5	5×10^4
Mexican blown asphalt:				
Penetration, 100	971	30×10^5	670×10^5	10×10^4
Softening point, 144 F.	95.5	20×10^5	1000×10^5	6×10^4
West Texas steam-reduced asphalt:				
Penetration, 89	971	4.6×10^5	7.0×10^5	7×10^4
Softening point, 114 F.	95.5	4.0×10^5	7.0×10^5	10×10^4
West Texas blown asphalt:				
Penetration, 84	971	13.2×10^5	27×10^5	10×10^4
Softening point, 118 F.	95.5	13.1×10^5	26×10^5	5×10^4
West Texas cracked asphalt:				
Penetration, 92	971	5.9×10^5	10×10^5	11×10^4
Softening point, 112 F.	95.5	5.8×10^5	9.2×10^5	6×10^4
California steam-reduced asphalt:				
Penetration, 97	971	11.7×10^5	11.7×10^5	0
Softening point, 110 F.	95.5	11.7×10^5	11.7×10^5	0
California blown asphalt:				
Penetration, 94	971	9.1×10^5	17.0×10^5	10×10^4
Softening point, 112 F.	95.5	11.5×10^5	17.0×10^5	4×10^4
Petroleum resin:				
Penetration, 86	971	8.5×10^5	8.5×10^5	0
Softening point, 107 F.	95.5	8.5×10^5	8.5×10^5	0
Vulcanized pure gum rubber	971	negligible	∞ [c]	323×10^4
	95.5	negligible	∞ [c]	323×10^4
Rubber-petroleum resin mixture vulcanized in place (The petroleum resin is the same as used above.)	971	7.8×10^5	∞ [c]	3.0×10^4
	95.5	8.2×10^5	∞ [c]	3.0×10^4

[a] Penetration at 77 F. per 100 g. per 5 sec. according to A.S.T.M. Method of Test D 5 – 25. Ring and ball softening point according to A.S.T.M. Method of Test D 36 – 26.

[b] n = a viscosity calculated from the initial speed; s = a viscosity calculated from the minimum or equilibrium speed; g = a coefficient of elasticity in shear; the ratio of stress to strain for a layer of unit thickness.

[c] In these instances continuous deformation is not possible.

The constants n, g, and s do not take account of creep or elastic after-effects. These slow secondary stages of deformation or recovery may be attributed to the extension and relaxation of portions of elastic structures which are associated with very high viscous resistances. For the more elaborate description of deformation behavior the constants n, g, and s may be supplemented by coefficients descriptive of these secondary effects. An extended mathematical development of the ideas here outlined will appear elsewhere.

Typical Values of Deformation Constants:

Table III gives values of *n*, *g*, and *s* for various asphalts, petroleum resins, and mixtures of rubber with petroleum resins, calculated from measurements made in the small open-end viscosimeter. Clay suspensions and gelatin have also been tested in this instrument. The values of *n* and *s* in Table III are independent of the shearing stress in most cases. Variations in *g* occur, in general, when deviations from viscous flow are small.

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RHEOLOGICAL INTERPRETATION OF ASPHALT TESTS

By R. N. Traxler,[1] H. E. Schweyer,[1] and J. W. Romberg[1]

Synopsis

The uses for asphalts are generally governed by their flow properties under application or service conditions. An evaluation of these properties in absolute units would be more satisfactory than the use of the numerous empirical tests now employed.

The types of flow encountered in asphalts are illustrated by rheological diagrams and terms employed in the rheology of asphalt are defined. A brief review is given concerning the colloidal nature of asphalt as related to its flow properties; methods employed and problems encountered in the investigation of the flow properties of such materials are discussed.

Data are given to show the relation between A.S.T.M. penetration test values at 77 F. and consistency in absolute units (poises) and the conclusion is drawn that this test is not satisfactory for the evaluation of rheological properties.

The rheological aspects of the ring-and-ball test are discussed and an equation relating consistency and ring-and-ball softening point for certain asphalts is given.

It is shown that high ductility is associated with a more or less narrow range of consistency depending upon the flow characteristic of the material being studied. This relationship between ductility and consistency is shown graphically. The physics of the ductility test are discussed and it is concluded that the test is too empirical to be helpful in the evaluation of the flow properties of asphalts.

Other empirical tests such as float, fluidity factor, and penetrations at high and low temperatures are also discussed as being inadequate for the quantitative evaluation of flow properties.

The consistency of asphalts showing complex flow must be determined at some standard rate of shear for which a tentative value of 0.001 reciprocal second is suggested. The degree of anomalous flow characteristics may be evaluated from measurements made at the standard rate of shear and one other suitable rate of shear. For such determinations the rotating cylinder viscometer is advocated.

The uses for asphalts are generally governed by their flow properties under application or service conditions. Recognition of the importance of measuring these characteristics in absolute units has been given in recent publications (**1**, **2**).[2] In general, empirical tests are used at present which are unsatisfactory because the combined results from several tests must be used in any attempt to evaluate flow characteristics. Also, such tests frequently lack the sensitivity required to differentiate asphalts satisfactorily, and the results are given in various arbitrary units which prevent satisfactory comparisons of data. A new empirical test method is often devised for each use, thereby resulting in a long and involved specification for a particular asphalt.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1194.

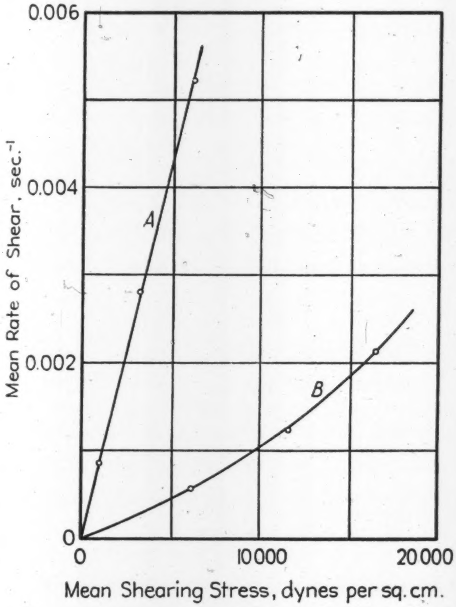

FIG. 1.—Rheological Diagrams for Asphalts A and B obtained at 77 F.

Evaluation of consistency in absolute (c.g.s.) units would simplify the problem, because it would eliminate the necessity for many of the tests now used in an attempt to measure flow properties.

Viscosity, η, is defined as the ratio of shearing stress F, to the rate of shear, S, and is given in poises where F is in dynes per square centimeter and S is in reciprocal seconds, or

$$\eta = F/S, \text{ poises} \quad \dots\dots\dots (1)$$

Rheological diagrams, which define the flow characteristics, are illustrated in Fig. 1. Data from which these curves were drawn are given in Table I. It is standard practice in constructing a rheological diagram, which is a plot of F *versus* S, to plot F as the abscissa.

If the ratio of F to S does not vary with F, the material is known as a *simple fluid to* or a *Newtonian liquid* and is said to exhibit true viscous flow (curve A of Fig. 1). The slope of the line is the fluidity in rhes which is the reciprocal of the viscosity in poises.

TABLE I.—FLOW DATA ON VISCOUS AND NONVISCOUS ASPHALTS.

Shearing Stress, dynes per sq. cm.	Rate of Shear, (sec.)$^{-1}$	Consistency at 77 F., poises ($\times 10^{-6}$)
Asphalt A—Vacuum Refined Venezuela Asphalt (117 F. ring-and-ball–95 penetration at 77 F. per 100 g. per 5 sec.)		
1 057	0.00083	1.28
3 332	0.00280	1.19
6 142	0.00524	1.17
Asphalt B—Air Blown Gulf Coast Asphalt (132 F. ring-and-ball–52 penetration at 77 F. per 100 g. per 5 sec.)		
6 160	0.00058	10.60
11 700	0.00126	9.26
16 400	0.00214	7.66

Data obtained by the falling coaxial cylinder viscometer

If the ratio of F to S varies with F, the numerical value for viscosity depends upon the rate of shear and the material is said to be a *complex fluid* or a *non-Newtonian liquid* (curve B, Fig. 1). In comparing the flow properties of complex fluids, consistency in absolute units must be determined at essentially the same rates of shear. The exact values for these rates of shear that should be used for asphalts are not known, although recent experiments have indicated their order of magnitude.

There are three general types of complex flow: (1) *quasi-viscous flow* (curve *B*, Fig. 1), in which the material deforms under any shearing stress, (2) *plastic flow* in which the material does not show permanent deformation until a critical shearing stress (the yield value) is reached after which it deforms so readily or quickly that, except for inertia effects, the stress never exceeds the yield value, and (3) *plastico-viscous flow*, in which no permanent deformation occurs at shearing stresses below the yield value after which the material deforms at a constant ratio of *F* to *S*, this ratio being called the plasticity.

In describing the rheological properties of materials, certain standard terms must be used. The term *viscosity* is by definition limited to simple fluids, as is *fluidity* which is the reciprocal of viscosity.

For plastico-viscous materials, the *mobility* in rhes is the slope of the line on the rheological diagram. The *plasticity* in poises is the reciprocal of the mobility. For other types of flow the term *consistency* in poises should be used. The terms viscosity and consistency are both used in this paper but the former is reserved for those materials which are essentially simple liquids. The reciprocal of consistency might be called the *pliability*, the unit of which is the rhe. Plasticity and consistency are analogous to viscosity while mobility and pliability correspond to fluidity.

Nellensteyn (3, 4) conceived asphalts as colloidal systems and suggested that each asphaltic micelle had a nucleus of carbon surrounded by absorbed asphaltenes, each successive layer being composed of hydrocarbons of lower molecular weight and higher hydrogen to carbon ratio. These micelles are considered to be kept in dispersion in the oily continuous medium (petrolenes) by the so-called asphaltic resins which serve as protective colloids or stabilizing agents. The only part of this theory that has not received general acceptance is the concept of a carbon nucleus for each micelle.

Pfeiffer and Saal (5) have elaborated on Nellensteyn's theory and have used the colloidal properties of various types of asphalt to explain their rheological characteristics. These authors believe that when the asphalt contains sufficient of the necessary constituents for the formation of the outer regions of the micelles, the asphaltenes are fully peptized and able to move as freely as the viscosity of the oily phase permits. "Such a system will show almost purely viscous flow, except for a marked intramicellar elasticity." These highly dispersed "sol" types of asphalt are simple liquids, giving rheological diagrams similar to curve *A* of Fig. 1.

However, if there is a shortage of asphaltic resins, the micelles will be subjected to a mutual attraction, varying in amount depending on the lack of dispersing agent. When such unprotected micelles approach each other, they will form a more or less firm bond, requiring the application of a force to separate them. The spaces in the "gel" structure formed by the incipient coagulation of the micelles are filled by the intermicellar liquid. Asphalts of the "gel" type give rheological diagrams of the type shown in curve *B* of Fig. 1, and exhibit all of the anomalous flow characteristics of complex liquids.

The transition from "sol" to "gel" or *vice versa* can be accomplished by lowering or raising the temperature of the asphalt. It is probable that this transition occurs over a rather wide range of temperature below the softening point of the asphalt. For certain asphalts which are non-Newtonian at 25 C., it has been found that when the temperature is raised so that the consistency decreases to the order of 200,000 to 1,000,000 poises, the bitumen becomes a simple liquid.

A series of asphalts made by a given process from a particular type of crude oil shows a transition from "sol" to "gel" type as the degree of processing is increased. In general, air-blown asphalts exhibit more anomalous flow properties than steam-refined asphalts, although the harder materials of the latter type exhibit flow characteristics similar to softer air-blown asphalts. Transition from simple to complex flow ("sol" to "gel" type) occurs at the following approximate consistencies obtained by processing:

Type	Viscosity at 77 F., poises ($\times 10^{-6}$)	Penetration at 77 F.	Ring-and-Ball Test, deg. Fahr.
Steam-refined....	7	40 to 50	130
Air-blown........	3.5	60 to 100	125

In addition to the rheological properties already discussed, asphalts exhibit *elasticity* (6), that is, the ability of a material to return to its original shape after deformation when the deforming stress is removed. This property is exhibited by the harder air-blown asphalts to a greater extent than by other types. The reciprocal of elasticity might be called *flaccidity*. The evaluation of elasticity in asphalts is such a complex problem involving shear, time of deformation, time of recovery, and method of measurement that at present no quantititive procedures are available.

Asphalts also exhibit *thixotropy* (6), the property that results in lower values for consistency if the material is worked under a constant shearing stress. Under continued working the consistency gradually approaches a constant value. Most asphalts exhibit this property to some extent but the harder asphalts are the most thixotropic.

The thixotropic and elastic properties of asphalts are the result of the formation of some type of structure caused by selective orientation of certain components of the asphalt. The rate at which this structure builds up is evaluated by age-hardening studies (7) wherein the increase in consistency with time is measured. This property is more pronounced in complex liquids and thus air-blown asphalts exhibit age hardening to a greater extent than do other types of bitumen. Some of the structure can be removed by continuous mechanical working for extended periods of time. Also, up to 90 per cent of the increase in consistency with time can be removed by remelting. It has been found (8) that the percentage rate of increase of consistency with time is not influenced by the presence of dispersed mineral powders.

Many methods for evaluating the change of consistency of asphalts with temperature have been proposed. Most of them are strictly empirical and are based on measurements of consistency that are not expressed in absolute units. Detailed reviews (1, 9) of the historical development of the various methods have been given. The Asphalt Viscosity Index, which is the percentage decrease in consistency (in poises) for a 1 C. increase in temperature, is one of the methods employing viscosity expressed in absolute units. If the consistency of the asphalt is about 10^6 poises, a 1 C. change will result in about 20 per cent change in consistency. A similar temperature variation for asphalts having a viscosity of about 10^4 poises results in a 12 per cent change in viscosity, while an asphalt having a viscosity of about 1 poise will change about 4 per cent in viscosity for a unit temperature change.

Presence of a mineral powder dispersed in an asphalt has no marked effect on temperature susceptibility expressed as percentage decrease in consistency per 1 C. rise in temperature (9).

The rheological characteristics discussed above may be considered as proof of the colloidal nature of asphalt. However, further and more direct evidence was obtained (10) from the appearance of asphalt surfaces treated by solvents (for example, 86° Baumé Naphtha or Ethyl Ether) capable of dissolving the petrolenes and resins or petrolenes alone. When examined under a microscope equipped with an attachment for viewing an opaque surface with oblique light, solvent-treated asphalts of the "gel" type gave distinct surface patterns, whereas those of the "sol" type gave no design discernible at a magnification of 220 times.

The empirical flow tests used at present do not evaluate quantitatively the many types of anomalous flow possessed by asphalts. In numerous instances selection of an asphalt suitable for a particular purpose depends upon some combination of properties which cannot be evaluated except by rheological methods. Therefore, a knowledge of rheological properties and interpretation of empirical test data in absolute units is essential to the satisfactory selection of asphaltic materials.

Apparatus have been described elsewhere (1, 11) for measuring the viscosity of asphalt over the range from 1 to 10^8 poises. Although the rotating cylinder viscometer is preferred because it offers the greatest flexibility in operation, the falling coaxial cylinder type is somewhat less expensive and more simple to operate and is satisfactory for use with Newtonian asphalt. The range of viscosity that can be measured by the latter viscometer is 10^4 to 10^7 poises, and the instrument is particularly adapted to a study of age-hardening characteristics.

Where the true viscosity cannot be measured because the asphalt is a complex fluid, it is necessary to establish a standard procedure for making all determinations at similar rates of shear (6). The conicylindrical rotating cylinder viscometer is more satisfactory for this purpose than any other type of instrument. As a first solution to the problem of selecting rates of shear for complex fluids, it is recommended that a mean rate of shear of 0.001 reciprocal second be used for viscosities up to the order of 10^8 poises at 77 F.

TABLE II.—FLOW DATA ON ASPHALTS FROM VARIOUS GULF COAST CRUDES.

Penetration at 77 F., 100 g. per 5 sec.	Ring-and-Ball, deg. Fahr.	Ductility at 77 F., 5 cm. per min., cm.	Consistency at 77 F., poises ($\times 10^{-6}$)
Steam and Vacuum Refined			
178	107	141	0.29
169	108	124	0.33
93	115	200+	1.17
90	118	200+	1.10
57	126	200+	4.10
53	127	200+	5.00
37	130	200+	8.7
33	137	200+	15.2
18	147	37	63.5
Air Blown Series I			
134	113	130	1.25
101	123	128	2.65
68	135	16	13.9
Air Blown Series II			
...	90	57	0.057
281	98	93	0.099
155	108	142	0.45
143	110	127	0.59
91	118	200+	1.63
70	125	192	4.50
56	132	169	7.50
48	134	118	15.8
42	142	43	22.8
36	145	12	49.5

Data obtained by the falling coaxial cylinder viscometer.

Consideration of the many types of flow characteristics possessed by asphalt indicates the necessity of using standardized techniques in measuring its consistency. The sensitivity of determinations made in absolute units makes it imperative that the samples are tested at the same age, under close temperature control, at similar rates of shear, and that care is taken not to change the asphalt in melting and filling the viscometers. A precision of better than 2 per cent can be attained under the most favorable conditions.

$$\eta = (5.13 \times 10^9)/P^{1.93} \ldots\ldots (2)$$

where

η = consistency in poises, and
P = penetration under a load of 100 g. for 5 sec.

It has been shown (14) that this relation is not valid for different asphalts if either the temperature of the test is changed or if the consistency is changed by processing.

In order to illustrate the relation between penetration and consistency expressed in absolute units, data are given in Table II. These are plotted in Fig. 2 together with the curve computed from Eq. 2 and data given in the literature (14)

These curves show that the results calculated using Eq. 2 are too low and, further, that air-blown asphalts have different consistencies from steam-refined asphalts of the same penetration. The type of crude oil source and the method of processing of air-blown asphalts have considerable effect on the position of the

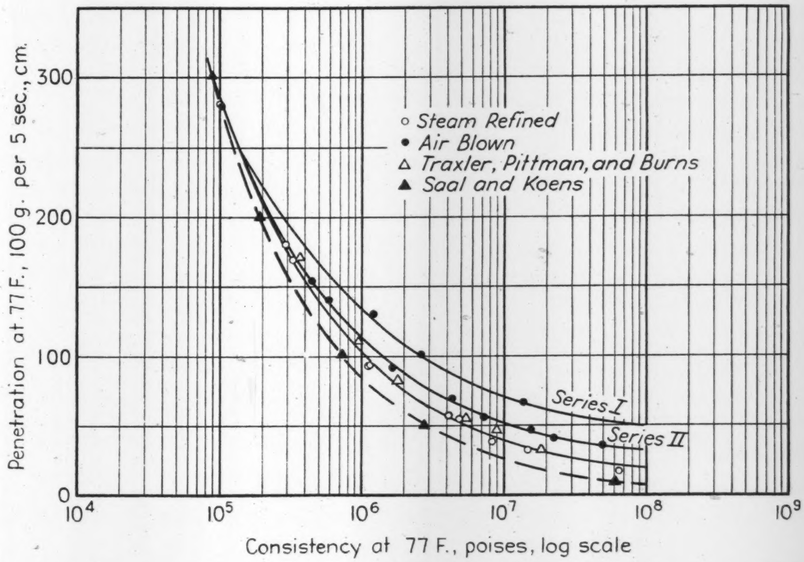

FIG. 2.—Penetration-Consistency Curves for Various Steam-Refined and Air-Blown Asphalts.

EMPIRICAL FLOW TESTS

The Penetration Test:

The A.S.T.M. penetration test[3] is not satisfactory for measuring consistency in absolute units either on a theoretical (12) or empirical basis because the test measures a combination of permanent and elastic deformation and adhesiveness of the asphalt for the steel needle. Saal and Koens (13) proposed a relation for viscous bitumens (those whose consistency does not vary with the applied shearing stress) which is as follows:

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[3] Standard Method of Test for Penetration of Bituminous Materials (D 5 - 25), 1939 Book of A.S.T.M. Standards, Part II, p. 412; Part III, p. 185.

curve as shown by series I and II of Fig. 2, where series I is more highly blown (blown from a lower starting consistency) than series II.

The curves shown for air-blown asphalt in Fig. 2 apply only to asphalts blown to the same degree as those used in these experiments since more highly blown products might show even higher penetrations at a given viscosity. This effect leads to the possibility that the difference in penetration of an air-blown asphalt and the curve for Newtonian asphalts of the same absolute consistency might be used to evaluate the loss in adhesiveness resulting from air blowing.

The curve in Fig. 2 may be used to estimate the consistency in absolute units of viscous steam-refined asphalts from the penetration. However, the curves for air-blown asphalts are specific with the type of asphalt, method of processing and rates of shear used in the consistency measurements. It should be noted that some points deviate considerably from the curves due to the lack of sensitivity of the penetration test.

It is interesting to note that the shapes of the curves in Fig. 2 are manifestations of the empirical nature of the penetration test. The A.S.T.M. penetrometer needle consists of a cylinder having a truncated conical end, the latter being 5.4 mm. long (equivalent to 54 penetration). The resistance to immersion offered by the conical section is much less per unit of penetration than that of the cylindrical section. Consequently, in the low penetration range, the viscosity must be increased greatly for each unit decrease in penetration as is shown by the flatness of the curves in Fig. 2. However, in the high-penetration range the relatively high resistance to immersion of the cylindrical part of the needle requires a smaller increase in viscosity for a unit decrease in penetration and the curve for this region has a large slope. There is a transition range connecting the high and low penetration ranges. The curvature in this region is dependent proportionately on whether the immersion of the conical part of the needle represents a large or small portion of the total distance of penetration.

The relatively high shearing stresses used in the penetrometer (2 to 150 times that used in the ordinary falling coaxial cylinder viscometer) results in flow determinations being run at very large rates of shear which vary with each unit of penetration. Based on the work of Thelen (**15**) and Rhodes and Volkmann (**16**), Mack (**17**) has suggested that the relative flow characteristics of asphalts can be evaluated by means of the penetrometer. However, from a study (**12**) on various kinds of asphalts, it was found that with the penetrometer flow is not laminar and that only a portion of the penetrated length of the needle is wetted by the liquid. Accordingly, it was concluded that such a method is unsatisfactory for evaluation of rheological properties.

The Ring-and-Ball Softening Point Test:

The A.S.T.M. ring-and-ball softening point test[4] measures the temperature at which the material being tested reaches a consistency such that it will have flowed a definite distance from the time heat was first added. The viscosity at this temperature has been shown (**11, 18**) to vary from 14,000 to 30,000 poises for asphalts. Rhodes and others (**19**) also reported results by Saal in which the viscosity at the ring-and-ball temperature for asphaltic bitumens varied from 8000 and 20,000 poises. In the same paper these authors show that for coal-tar pitches the viscosity at the ring-and-ball temperature may vary from 9000 to 27,000 poises. Although it has been stated in the literature (20) that the ring-and-ball softening point represents a definite viscosity, it is concluded from the above results that this is not true.

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[4] Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 - 26), 1939 Book of A.S.T.M. Standards, Part II, p. 417.

Figure 3 shows consistency in poises at 77 F. plotted against the ring-and-ball softening point for the various Gulf Coast asphalts given in Table II. There appears to be good agreement for most of the data with no systematic deviation for either steam-refined or air-blown asphalts. However, for vacuum or steam-refined asphalts having very high temperature susceptibilities, the viscosity values read from the curve will be lower than are obtained experimentally.

The equation for the empirical relation shown in Fig. 3 is given by Eq. 3, the constants of which were determined by the method of averages:

$$\log \eta = 0.0543\,M - 0.232 \ldots (3)$$

where M = ring-and-ball softening point in degrees Fahrenheit.

This equation gives calculated values for consistency within plus or minus 17 per cent of the experimentally determined data. This may be considered fairly good agreement since the allowable error in the ring-and-ball test is plus or minus 1 F. which corresponds to about a difference of plus or minus 14 per cent in consistency based on Eq. 3. These rela-

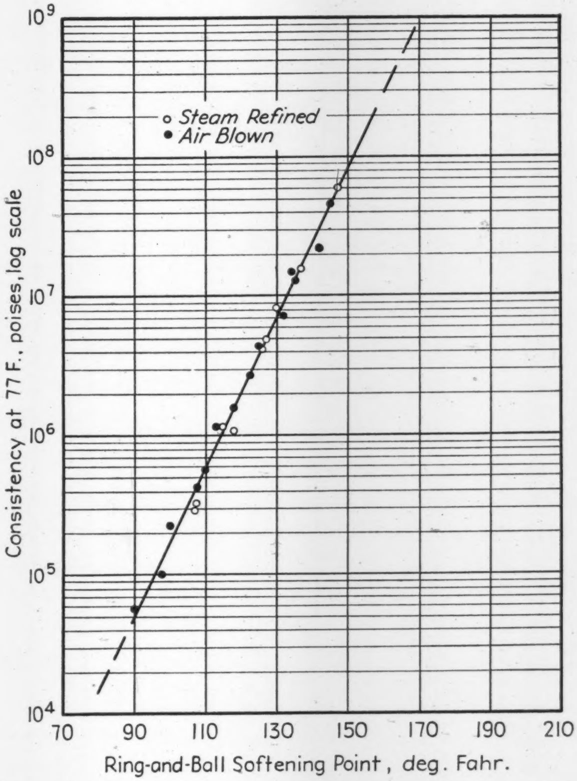

Fig. 3.—Ring-and-Ball - Consistency Relation for Various Steam-Refined andAir-Blown Asphalts.

tively large deviations are attributed to lack of sensitivity of the ring-and-ball method of test and in a smaller degree to experimental error in both the ring-and-ball and consistency measurements.

The formula given in Eq. 3 may be used to estimate the consistency in absolute units for asphalts, but it should be recognized that deviations up to about 30 per cent are possible. In using Eq. 3 for non-Newtonian material it must be assumed that it applies only at the mean rate of shear used in this study (0.001 reciprocal second). The empirical conversion of ring-and-ball softening point to absolute consistency, while satisfactory for the ranges shown in Fig. 3, is not recommended as a substitute for viscometer measurements because the limits of the empirical relationships are not known and, as mentioned above, the relationship does not hold for vacuum or steam-refined asphalts of very high temperature susceptibility.

The relatively good agreement for air-blown asphalts shown in Fig. 3 is an indication that the rate of shear used in this study is satisfactory for determining the consistency of such materials in absolute units. The selection of a numerical value for rate of shear at which the viscosity of non-Newtonian materials should be evaluated has been a problem in the past and the results of this study offer a first solution. It is possible that future work on this subject may indicate modifications in the rate of shear used or in the numerical values of the constants in Eq. 3.

The fact that the viscosity is not constant at the ring-and-ball temperature might be expected from a consideration of the physics of the test. In the first place the ring-and-ball method combines the principles of a falling ball viscometer and those of the falling coaxial cylinder viscometer since the ball falls through the asphalt (falling ball effect) but at the same time moves the mass of asphalt in a shearing action similar to the falling coaxial cylinder. (As a matter of interest if the apparatus is considered as a falling coaxial cylinder viscometer where the ball is assumed as a cylinder the shearing stress at the walls is 135 dynes per sq. cm.) In the second place the rate of shear varies in the ring-and-ball method and as has been noted previously any variation in the rate of shear will affect the numerical value of the viscosity for non-Newtonian liquids. While it may be argued that all asphalts at the ring-and-ball temperature are simple liquids, it is, nevertheless, true that at the start of this method of test the asphalt is subjected to shear at temperatures where the viscosity is many times greater than that at the ring-and-ball temperature and at which the material may be non-Newtonian.

The Ductility Test:

The A.S.T.M. ductility test[5] does not evaluate consistency because the test conditions (shearing stress and rate of shear) vary with the flow characteristics of the material being studied. The precision of the test is low with duplicate tests sometimes varying up to 50 per cent although the standard testing procedure stipulates an allowable variation of only 10 per cent.

A plot of ductility *versus* consistency is given in Fig. 4, which shows that ductility is related to consistency in absolute units. Data were taken from Table II. At low consistencies, asphalts have low ductility values partly because they flow out of the mold too rapidly. However, as the consistency increases a region of high ductility is reached. As the consistency becomes still greater a decrease in ductility results because the flow properties of the asphalt do not allow it to deform sufficiently rapidly at the high shearing stress employed and the thread breaks.

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[5] Standard Method of Test for Ductility of Bituminous Materials (D 113 - 39), 1939 Book of A.S.T.M. Standards, Part II, p. 398.

As shown in Fig. 4, air-blown asphalts tend to give lower ductilities than steam-refined asphalts of the same viscosity. For this reason and also because of the low sensitivity of the ductility test there is a range of ductility, shown in the shaded portion of the graph, corresponding to a given consistency expressed in absolute units. The upper limit of the shaded portion at any given viscosity may be considered to be the maximum ductility for steam-refined asphalts. The lower limit of the shaded portion is based on data from series I in Fig. 2 and two additional experimental points discussed later. This lower limit may be considered the minimum ductility for normal air-blown asphalts whose consistency is determined at a rate of shear of 0.001 reciprocal second. There may be cases where the ductility of peculiar blown asphalts of any given consistency will not fall with in the shaded areas, but the curves shown may be considered to be generally applicable.

The curves shown in Fig. 4 apply to data at any temperature and show how changing the temperature of test will affect the ductility values through the influence of temperature on consistency. In order to illustrate the validity of the chart the following experiments were conducted. The consistencies of two asphalts, C and D, from series II of Fig. 2 were determined at 55 and 77 F. and the ductilities at 77 F. (5 cm. per min.) with the following results:

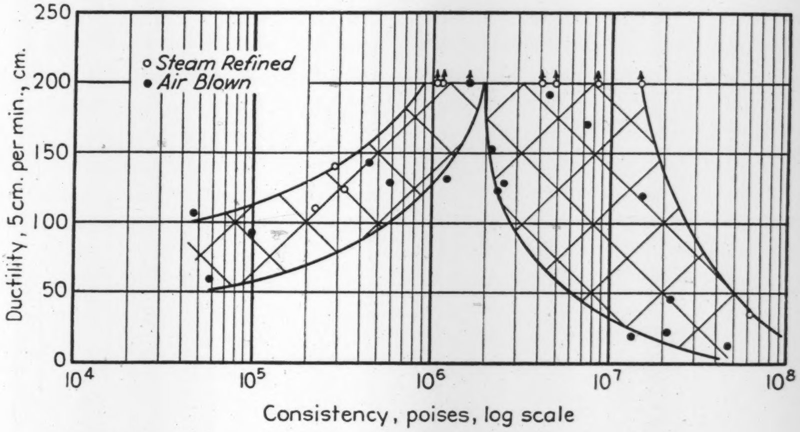

FIG. 4.—Variation of Ductility of Asphalts for a Given Absolute Consistency.

	Consistency, poises ($\times 10^{-6}$)		Ductility at 77 F., 5 cm. per min., cm.
	77 F.	55 F.	
Asphalt C........	0.099	2.42	93
Asphalt D........	1.63	35.2	200+

Reference to Fig. 4 shows that if the temperature of the ductility test is lowered to 55 F., the ductility of asphalt C should increase to 200+ and that of asphalt D should decrease to less than 80. Ductility values obtained experimentally at 55 F. were 200+ for asphalt C and 77 for asphalt D.

In a similar manner, a *highly* blown asphalt having a consistency of 21.0 × 10^6 poises at 77 F. had a ductility of 22 at 77 F. The consistency of this material at 96 F. was 2.3 × 10^6 poises which would indicate (Fig. 4) a ductility at that temperature of 130; experimental determination at 96 F. gave 124 cm. These data, obtained on highly blown asphalts (not shown in Table II) indicate the necessity for the wide range of ductility at a given consistency shown in Fig. 4.

The chart shows that the region of high ductility for steam-refined asphalts extends over a larger range of consistency than for air-blown asphalts. This may be attributed to the flow properties of the latter since it will be shown below that the more anomalous the flow characteristics the smaller is the range of consistency over which high ductility can be attained. For truly viscous asphalts, very high ductilities may be obtained over a range of from about 1 × 10^6 to 15 × 10^6 poises corresponding to a ring-and-ball softening point range of about 115 to 135 F.

In the ductility test the force required to pull a soft asphalt out into a long thin thread is small compared with the force required to break a high consistency material having a low ductility. However, the actual physics of the test are difficult to explain. One explanation for the necessity of showing a range of ductility for a given viscosity has been noted in the literature (21). It was stated for non-Newtonian fluids that if the consistency decreases too rapidly with increasing shearing stress (which occurs as the cross-section of the thread decreases) there occurs an "unproportionately rapid deformation—which may cause necking." Once this effect is started it is cumulative and the thread soon breaks. Since the steam-refined asphalts can attain higher consistencies than air-blown asphalts before these non-Newtonian effects occur, they may be processed to a higher consistency than air-blown asphalts before the ductility begins to decrease. These effects account for the range of ductility shown in Fig. 4 for high consistencies. No explanation is advanced for the range of ductility at low consistencies other than the fact that air-blown asphalts show more anomalous flow properties than steam-refined ones which may be exhibited also in this region of low consistency.

For asphalts showing high ductility—thereby indicating true viscous flow—the speed at which the ductility test is run should not change the results, since the rate of shear has no effect on the viscosity. However, for asphalts having low ductility (in the high-consistency range) an increase in the machine speed (increase in rate of shear) should accentuate the necking effect and give even lower ductilities. Exactly these results are indicated from an analysis of data given by Broome (1) and quoted in Table III. The data show that if the consistencies of asphalts B and C are decreased by raising the temperature sufficiently to remove the non-Newtonian flow effects, the ductility becomes independent of the machine speed. However, at lower temperatures for the same asphalts where non-Newtonian flow probably prevails, an increase in machine speed causes a decrease in ductility value.

TABLE III.—TYPICAL DUCTILITIES AT VARIOUS SPEEDS—AFTER BROOME (1)

Asphalt	25 C. (77 F.)			35 C. (95 F.)			45 C. (113 F.)		
	1 cm. per min.	5 cm. per min.	25 cm. per min.	1 cm. per min.	5 cm. per min.	25 cm. per min.	1 cm. per min.	5 cm. per min.	25 cm. per min.
B ...	2.5	Nil	Nil	115	26	Nil	150	124	145
C ...	45.6	26.5	11.0	150	75	56	140	145	150

A similar effect was found for a highly blown asphalt having a consistency of 21.0 × 10^6 poises at 77 F. and a ductility of 22 cm. at a speed of 5 cm. per min.

When this speed was reduced to 1 cm. per min., the ductility value was 88 cm., indicating that at the lower rate of shear the ductility for this highly blown material was more nearly like that for Newtonian asphalts.

These results indicate that the use, in the ductility test, of particular (low) rates of shear (depending upon the consistency) would yield similar results for non-Newtonian and truly viscous asphalts. At such rates of shear, similar ductilities would be obtained for all asphalts at a given consistency and the shaded portion of Fig. 4 would be reduced by a shift of the lower limit for ductility toward that for viscous asphalts.

It is apparent from these studies of the rheological aspects of the ductility test that, while it measures some combination of flow properties, it does not measure any specific property. The test cannot be considered as a measure of adhesiveness which is indicated more definitely by penetration-consistency relations. Further, the ductility test does not evaluate elasticity since blown asphalts, which show the greatest elastic effects, have the lowest ductilities.

The above facts are corroborated by Baskin (22) who stated: "I feel justified in stating that ductility is positively the most misleading test we have on record and has done more towards imbuing people with erroneous notions regarding asphalt than any other test of questionable nature."

Other Empirical Flow Tests:

A number of other empirical tests are used by asphalt technologists to evaluate the flow properties of asphalt. Among these are penetration and ductility at temperatures other than 77 F., float test[6] and fluidity factor (23). The purpose of these tests is the same, namely, to obtain information concerning the effect of temperature on consistency. Since each test is empirical and cannot be converted to consistency in absolute units, the results are meaningless by themselves and can be interpreted only in conjunction with results from other tests.

Discussion

With the exception of the ring-and-ball softening point test for certain asphalts, the empirical flow tests in common use cannot be used to evaluate the flow properties in absolute units with any degree of precision. Substitution of rheological methods for these empirical tests would simplify laboratory testing for both consumer and producer since less complicated specifications would be necessary.

For example, the use of three or four measurements of viscosity over the range from service to application temperatures would determine the effect of temperature on flow properties. Such determinations in absolute units define the flow characteristics quantitatively and do not require comparisons with results from other tests.

Measurement of the viscosities of the softer asphalts and of the harder materials which exhibit viscous flow is a relatively simple procedure. The determination of the rheological properties of asphalts that are complex fluids is, however, somewhat more involved.

First, in order to compare the consistencies of different asphalts showing various degrees of anomalous flow, measurements must be made at some standard rate of shear. Based on the correlation shown in Fig. 3 for asphalts exhibiting simple and complex flow, 0.001 reciprocal second appears to be a satisfactory rate of shear at which to compare the consistencies of asphalts showing anomalous flow characteristics. This rate of shear could be used conveniently, in an instrument of proper design, for asphalts having consistencies up to the order of 10^8 poises at 77 F. which correspond to penetrations of about 30 to 50 at 100 g. for 5 sec.

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[6] Standard Method of Float Test for Bituminous Materials (D 139 - 27), 1939 Book of A.S.T.M. Standards, Part II, p. 409.

Second, evaluation of the degree of anomalous flow exhibited by certain types of asphalts necessitates measurement of consistency at different rates of shear. Most asphalts exhibiting complex flow are of the quasi-viscous type. Accordingly determinations made at two rates of shear establish the rheological diagram if all curves pass through the origin. If the determination of the consistency at a rate of shear equal to 0.001 reciprocal second is always made as mentioned above, the degree of anomalous flow may be evaluated by making one additional consistency measurement at some other rate of shear. For asphalts possessing consistencies below 10^7 poises (above 50 to 70 penetration) at 77 F. a rate of shear of 0.01 reciprocal second probably would be satisfactory; for harder asphalts a rate of shear of 0.0005 is suggested. The relative consistencies at two rates of shear might be used as a quantitative measure of the degree of anomalous flow exhibited by various asphalts.

It should be noted that equilibrium consistencies must be used for all of these evaluations and comparisons of complex asphalts, that is, the asphalt must be subjected to the particular rate of shear until the shearing stress (and the calculated consistency) becomes constant.

All of the measurements of consistency for both viscous and quasi-viscous asphalts could be made in a rotating cylinder viscometer of such construction as to attain the desired shearing stresses and rates of shear. Interchangeable parts could be used for application to process control to insure speed in obtaining results and a relatively simple technique would be required.

All of the uses for asphalts are dependent in part on their flow properties; from both a practical and scientific point of view these properties should be evaluated quantitatively in absolute units in order to simplify laboratory testing.

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DISCUSSION

Mr. Eugene C. Bingham[1] (*presented in written form*).—Our Society has many methods concerned with the flow of materials. The designations often do not disclose whether the designers of the methods intended to measure flow or not. A penetrometer test may be imagined to be a measure of flow, a softening temperature is less obviously a measure of flow, and the volume shrinkage of clay on drying, or the tensile strength or the dye absorption of clay—seriously proposed for measuring the consistency of clay—would seem at first to be only very distantly related to flow.

Our empirical tests need to meet three requirements:

1. If they are devised to measure flow, they should measure that property and no other.
2. Our different methods should be capable of correlation. This is difficult, if not impossible, when one method expresses its results as a *distance*, another as a *temperature* and another as a *time*.
3. Our empirical tests are in no case expressed in the fundamental units of flow. This in itself is a fatal defect and an entirely unnecessary one. It should take no more time or effort to use the proper units of flow. Therefore all methods for measuring viscous or plastic flow should be expressed in absolute units.

What the particular method is, is a matter of no importance to us here and might be left to the convenience of the user, so long as it is capable of giving a precise measure of flow in the units desired.

We may offer a paeon of praise to Messrs. Traxler, Schweyer, and Romberg that they have already conceived of this problem in its entirety, and for one class of material have carried it steadfastly forward to so successful a conclusion. Their paper is much more than destructive criticism. They, and all of us, are indeed sorry to know that our old and highly respected methods are subject to grave limitations. Those tests we have inherited from days of small production, limited capital investment and inadequate scientific control. But the Jacob's staff has given way to the surveyor's transit. In days of mass production, of elaborate control and research laboratories with single contracts involving hundreds of thousands if not millions of dollars, it seems at least justifiable to know *when* we are measuring flow, and *how* it may be done. The authors, working with what some have regarded as an unpromising material to classify, are far from despairing; they are telling us how they believe it can be done.

The problem is far from done. There are those who will ask how we are to measure the plasticity of a given material and we may in all honesty confess that we do not know. But we will try. According to the motto of the immortal and always hopeful Lafayette, *"Cur non."*

Mr. W. F. Fair, Jr.[2] (*by letter*).—Although statements are made, with supporting references, that the viscosity at the temperature of the ring-and-ball softening point is not constant, the evidence given in this paper shows that, for certain materials at least, the viscosity at the softening point temperature must be approximately the same, else the agreement represented by the data of Fig. 3 would not have been found.

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[2] Industrial Fellow, Mellon Institute of Industrial Research, University of Pittsburgh, Pittsburgh, Pa.

That some other materials might not agree with the relation derived can be due to different temperature susceptibilities, as well as to lack of constant viscosity at the softening point temperature, since the consistencies reported have been determined at 77 F., which is lower than the softening point temperatures in the cases given. Inasmuch as there are conflicting data on this subject, it might be interesting to redetermine carefully the temperature susceptibilities, and the viscosities at the temperature of the softening points of typical bituminous materials.

The writer discussed this paper with Mr. E. W. Volkmann of Koppers Co. who agreed that the use of such terms as "pliability" and "flaccidity" might not be wisely introduced at this time when there still appears to be considerable disagreement about rheological definitions.

The general discussions in this paper, the points stressed, and the emphasis placed on the necessity of obtaining absolute rheological data, in preference to empirical tests which usually measure more than one property and are therefore frequently misleading, have all been well presented, and this pertinent contribution should aid materially in furthering the adoption of absolute viscosity standards for bituminous materials.

MR. C. M. BASKIN[3] (*by letter*).—If it were possible to displace the three outstanding yet perplexing test methods—penetration, ductility, and softening point—by the evaluation of flow properties directly, it would be the greatest advance made to date in defining and controlling quality of asphalt cements. Anyone concerned with asphalt technology needs little convincing that the penetration, softening point, and ductility tests are faulty—especially the latter—not only because these tests do not really define any property in particular, but also because of the numerous variables involved if we were to make these empirical tests cover any appreciable range of asphalts.

It would be a tremendous aid in process control if we were able to use one test with one variable to cover the whole range of asphalts. Up to now, we actually must run four tests—penetration, softening point, ductility, and viscosity—to tell whether a batch of asphalt is completed and fit for use. Viscosity is, no doubt, the most enlightening of our tests; but, because of inadequate equipment, our present viscosity determinations are limited in scope.

Consequently, if a whole range of asphalts could be tested in one and the same apparatus, and in this apparatus obtain flow properties, rather than something that is a jumble of a whole lot of properties, it would then simplify control to an amazing degree.

The possibilities of simplifying definition and classification of asphalts on the basis of flow properties, hence simplifying the process of selecting asphalt for any particular use, would certainly be a marked advance in the field of asphalt technology. Here, determination of flow properties may very likely furnish us with data that could be translated into terms of quality. I see no reason why flow determinations will not give us a true measure of temperature susceptibility.

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[3] Asphalt Technologist, Imperial Oil Limited, Toronto, Ont., Canada.

If the authors' objective is to develop a method of testing asphalt that will replace penetration, softening point, ductility and the conventional methods of viscosity determinations, fluidity factors, etc., one must keep in mind that any developed method will have to be given the same meaning as the combined results of the present empirical tests have to both the asphalt producer and user. There is quite a difference between saying that a test is faulty and that it is useless. I am not inclined to say that even identification tests are entirely useless. The present empirical tests, therefore, have considerable utility even though arriving at some logical conclusion usually requires a roundabout way.

Take, for instance, temperature susceptibility. I happen to be concerned with paving work in two extreme territories—in tropical regions and in sub-Arctic regions. Faulty as temperature susceptibility determinations are by means of the penetrometer, it has still given us a means of control which actually worked in practice. We have, for instance, definitely established that using lower susceptibility asphalt cement (susceptibility determined by penetrometer) materially reduces and often eliminates cracking of sheet asphalt pavements. Lowering susceptibility of the asphalt cement, so that for any given penetration at 77 F. it will show a higher penetration at 32 F. and a lower penetration at 100 F., made it possible, in a number of instances, to use a higher penetration asphalt cement at 77 F. to start with. What we were after is quite obvious, namely, to incorporate in the aggregate an asphalt cement that will have perceptible pliability at low temperatures, and yet not be too fluid at summer temperatures.

The penetrometer as a tool to measure temperature susceptibility is admittedly faulty, but it served our purpose. We managed, with this faulty testing method, to put down sheet asphalt pavements that do not crack at low temperatures nor distort at high temperatures. I do not mean to say that cracking of sheet asphalt pavements will be entirely prevented by so simple an expedient as reducing temperature susceptibility. There are a number of other factors that come into the play, principally aging or progressive hardening of the asphalt cement. Will determination of flow properties throw any definite light on this phenomenon? Will it be possible, by determining flow characteristics, to tell whether an asphalt will remain stable or will continue to change?

It therefore appears to me that the first step, at present, would be to develop a test method for asphalt flow properties that would replace penetration, softening point, ductility, and current viscosity tests, and show that this one test can be used for control and classification of an appreciable range of asphalts.

The next step would be to give flow property test results the same meaning as is ascribed to the penetration, softening point, and ductility tests. If this were possible, it would be an excellent start in the right direction, and a basis for the continuation of more sound development of asphalt testing and control.

MR. C. MACK[4] (*by letter*).—The authors are to be congratulated upon their efforts to eliminate empirical flow tests for asphalts. They propose to measure the consistency of asphalts by means of an apparatus which allows the consistency to be expressed in absolute units. For this procedure they chose an arbitrary rate of shear of 0.001 reciprocal sec. The relative consistencies at two given rates of shear are suggested as a quantitative measure of the degree of anomalous flow of quasi-viscous asphalts. Actually, however, this relationship does not define the flow property of such asphalts.

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[4] Research Chemist, Imperial Oil Limited, Sarnia, Ont., Canada.

Viscous flow is expressed by:

$$\eta = F/S \quad \ldots\ldots\ldots\ldots (1)$$

The flow property of such an asphalt is completely defined by one constant η.

Quasi-viscous flow is represented by:

$$(\eta) = F^n/S \quad \ldots\ldots\ldots\ldots (2)$$

This relationship requires the two constants (η) and n to define the flow property. The constant (η) has the dimensions of viscosity, but is different from η, and represents the consistency at a shearing stress equal to 1. If n is equal to 1, Eq. 2 is the same as Eq. 1. If n is larger than 1, the consistency decreases with increasing shearing stress. Quasi-viscous asphalts of the same consistency at a given shearing stress are the more resistant to flow at lower stresses the higher the value of n. This constant therefore serves as a numerical value for the degree of anomalous flow and can be obtained from the relationship:

$$n = (d \log S)/(d \log F) \quad \ldots\ldots (3)$$

The authors criticize the penetration test as not being satisfactory for measuring consistency in absolute units. They present data in their Table II to illustrate that the penetration-consistency relationship varies with the processing method of the asphalts.

An analysis of the data shows that their criticism is not justified. For example, the asphalts of 281 and 36 penetration of the air-blown series II have consistencies of 9.9×10^4 and 49.5×10^6 poises. At a proposed rate of shear of 0.001 the corresponding shearing stresses are 99 and 49,500 dynes per sq. cm., whereas the shearing stresses applied during the penetration determinations are considerably higher and are approximately 1.2×10^5 and 1.5×10^6 dynes per sq. cm. Since the consistency of quasi-viscous asphalts changes with the shearing stress as shown in Eq. 2 above, it is essential that the consistency determinations be carried out at shearing stresses equivalent to those of the penetrations; otherwise no comparison can be made between penetration and consistency.

MESSRS. R. N. TRAXLER,[5] H. E. SCHWEYER,[5] AND J. W. ROMBERG[5] (*Authors' closure by letter*).—In replying to Mr. Fair's comments it might be emphasized that the empirical nature of the ring-and-ball softening point procedure is such that no conclusion may be drawn from it regarding rheological characteristics of different types of materials unless the temperature-viscosity characteristics of the material are established. The data illustrated in Fig. 3 were intended to show only the order of magnitude of consistency for asphalts of certain softening point ranges because of the empirical nature of the relationship and its limitations. The relation implies only that all asphalts of the same ring-and-ball softening point have the same consistency at 77 F. Their viscosities at the ring-and-ball temperature can be identical only if their temperature-viscosity susceptibilities are the same, which is not to be expected.

In replying to Mr. Baskin's discussion it should be emphasized that the transition from empirical tests to absolute determinations is necessarily slow and must pass through a period during which both types of tests are used. However, when absolute determinations are made by a sufficient number of independent investigators, the empirical tests will become obsolete with benefit to both producer and consumer.

Mr. Mack's discussion of the evaluation of the flow of non-Newtonian asphalts is pertinent and emphasizes the necessity for obtaining rheological data on such materials.

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[5] Director of Asphalt Research, Chemical Engineer, and Chemist, respectively, The Texas Company, Port Neches, Texas.

It was stated in the paper that the measurement of the flow properties of non-Newtonian asphalts consists of two parts: first, a consistency measurement at some standard rate of shear, and second, an evaluation of the degree of anomalous flow. It was not suggested that the flow property of non-Newtonian materials could be evaluated by determining the degree of anomalous flow alone. Actually, the evaluation of the flow properties of complex liquids is more involved than merely fitting the data to an equation such as Mr. Mack's Eq. 2.

First, the consistency measurements must be made in an apparatus that yields results in absolute units which are not subject to theoretical criticisms; second, the form of the equation may be more complex than a simple power equation[6,7,8]; and third, in explaining the behavior of different asphalts, the practical results must warrant the use of a complicated treatment.

The application of the rheology of asphalts to practical problems requires the most simple techniques. However, theoretical objections to certain procedures cannot be overlooked in order to attain simplicity, otherwise the advantages resulting from the use of rheological methods will be vitiated. Thus, the penetrometer, while simple to operate, requires a complicated mathematical treatment as has been pointed out recently by Mill and Harrison.[9] The penetrometer is more limited in range and is subject to more criticisms than properly designed viscometers that yield results in absolute units over a wide range of viscosity. These factors and considerable research were the basis for the suggested methods of evaluating the degree of anomalous flow. However, it was not intended that the suggested procedures should be the final answer since future investigations may result in more satisfactory methods for evaluations.

The shearing stresses attained in the penetrometer are considerably higher than were used in the viscosity determinations and if higher stresses could have been used (as suggested by Mr. Mack) the curves for series I and II, in Fig. 2 would have been displaced downward. However, such procedures are impossible in measuring the viscosity of relatively fluid materials by the falling coaxial cylinder method, and they are practically impossible by other absolute methods. In addition, there is no way of knowing what shearing stress to select since it would vary with each depth of penetration. These considerations indicate the impracticability of constant shearing stress measurements, but of greater importance is the theoretical advantage of comparing flow measurements on non-Newtonian asphalts at constant rates of shear. Flow is a manifestation of shear. While it is true that the shearing stress produces the shear in a non-Newtonian asphalt, the rate of shear, under the conditions of stress, is an intrinsic property which determines the flow of the asphalt. Therefore, in comparing *flow properties* of such materials it is more logical to use a basis of equal rates of shear, as has been suggested previously,[10] rather than a basis of equal shearing stresses.

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A STUDY OF THE RECOVERY OF THE BITUMEN FROM SOFT ASPHALT CEMENTS BY THE BUSSOW METHOD

By Carl Bussow[1]

Synopsis

With an increasing number of State specifications requiring the extraction and recovery of the asphalt from paving mixtures in order to control the physical properties of the asphalt actually in the pavement, a simple and reliable method for the recovery of the extracted asphalt should be available. The author's method is described and data are presented on the recovery of nine asphalts from different sources, ranging from 50 to 270 in penetration, and showing the influence of the maximum temperature of recovery on the penetration and loss on heating of the recovered asphalts. Precautions necessary in the extraction and recovery of asphalts by either the author's method or the Abson method are discussed. An explanation is offered for the loss of ductility in asphalts recovered by the Abson method.

Since the publication, in 1936,[2] of the author's method of recovering bitumen from paving and waterproofing materials, many laboratories, including the Asphalt Institute,[3] have used it and found it to be both simple and reliable for asphalt cements ranging up to 88 penetration at 77 F. (25 C.). When the method was applied to much softer asphalts appreciable losses in penetration occurred. The author decided to investigate the effect of varying the maximum temperature of distillation upon the penetration of the recovered asphalt. The description of the Bussow method for the recovery of asphalt cements is given below.

Apparatus:

The apparatus consists of a 500-ml. short-neck flask similar in design to the flask used in the Society's Standard Method of Test for Distillation of Tar Products Suitable for Road Treatment (D 20 - 30).[4] The regular D 20 flask may be used but the larger flask permits better control of foaming. The flask is connected to a water-jacketed condenser and a convenient receiver to collect the distillate. The thermometer is placed with the bulb $\frac{1}{4}$ in. (6.5mm.) above the bottom of the flask, which is not shielded.

Procedure:

The bituminous material is extracted with hot benzol in a convenient apparatus, preferably of metal construction to prevent photochemical effects. The benzol extract, freed of fine mineral aggregate by centrifuging, is placed in the flask and distilled at the rate of 5 ml. per min. The small flame is placed toward the side of the flask at the beginning of the distillation to produce circulation of the liquid and to reduce foaming as the viscosity of the liquid increases. Should the foaming become excessive and rise too close to the neck of the flask, a flame played on the sides of the flask will cause the foam to subside. At some temperature between 150 to 200 C., depending on the fluidity of the asphalt, foaming will practically cease. At this point the flame is moved directly under the flask and the heat increased so the asphalt will attain a temperature of 300 C. in 5 min. The distillation is then stopped and the asphalt is immediately poured into a deep 4-oz. tin container, or any other suitable size, resting upon a heat-insulating substance, to prevent rapid cooling of the bottom layer of the residue. The asphalt is stirred slowly with the thermometer until it cools to 160 C. or until it fumes only slightly; it is then ready to be poured for tests on the bitumen.

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[1] Vice-President and Chief Chemist, A. W. Dow, Inc., New York, N. Y.

[2] Carl Bussow, "Notes on a Method of Recovering Bitumen from Paving Materials," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 7, January 23, 1936, p. 160.

[3] Prévost Hubbard and Henry Gollomb, "The Hardening of Asphalt with Relation to Development of Cracks in Asphalt Pavements," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 9, December 8, 1937, p. 165.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 390.

The selection of the above-stated time, temperature, and rate of distillation is arbitrary. The rate of distillation is that ordinarily used in standard distillation tests. The time required for the final heating to 300 C. may be extended to 10 min. without affecting the result. The maximum temperature of 300 C. was ascertained by methods of trial and error.

Precautions:

There are several precautions necessary in extracting and recovering asphalt from commercial materials.

1. It is essential that the insoluble material be washed absolutely clean of asphalt so that the final benzol washing be not discolored. Asphalt is selectively adsorbed by many otherwise inert materials and failure to observe this obvious precaution often gives a softer residue through loss of the harder and less soluble fraction of the asphalt.

2. Finely divided insoluble matter should always be removed from benzol solution in a centrifuge as the effect of small quantities of foreign material on the ductility of asphalt is very marked.

3. The benzol used for extraction should be pure and should have a distillation range of less than 2 C. Commercial benzols often contain toluol or even xylols and should be tested before using.

4. The size of the distillation flask should bear some relationship to the quantity of asphalt being recovered. The author has known of attempts to recover 10 or 15 g. of asphalt in a 500-ml. flask instead of using a flask or retort of 100 to 150-ml. capacity. Usually the quantity of asphalt recovered lies between 50 and 100 g.

5. While it is essential in using the Abson method[5] to extract and recover the asphalt within 10 hr., with the author's method of recovery the asphalt may remain in solution for days, or even weeks, before it is recovered.[6]

6. If the asphalt contains free carbon or other insoluble matter caused by excessive cracking in its handling, the recovered asphalt cannot be compared with the original asphalt, as such insoluble matters have a marked effect on the penetration and ductility of the asphalt.

Effect of Maximum Temperature of Recovery:

In this investigation nine asphalts ranging from 49 to 270 in penetration at 77 F. (25 C.) were dissolved in hot benzol and recovered in the standard D 20 flask. With the exception of asphalt No. 9, as shown in Table I, the penetration of the residue when the distillation was stopped at 225 C. was slightly higher than in the original asphalts. At maximum recovery temperature of 250 C. the penetrations could be considered checks of the penetration of the original asphalts as the maximum variation, except for No. 9, was only 6 points; with asphalt No. 9 the recovered asphalt was 18 points (6.7 per cent) lower in penetration than the original asphalt.

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[5] Gene Abson, "Method and Apparatus for the Recovery of Asphalt," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 704 (1933).

[6] V. B. Steinbaugh and J. D. Brown, "A Study of Asphalt Recovery Tests and Their Value as a Criterion of Service Behavior," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 9, December 8, 1937, p. 138.

The loss on heating for 5 hr. at 163 C. and the penetration after heating are shown for the original and the recovered asphalts. In Table I, the loss on heating of all asphalts recovered at 250, 275, and 300 C., agrees closely with the loss on heating on the original asphalt cements, the maximum deviation being only 0.2 per cent. The penetration of the residue after heating also agrees with the results on the original asphalt, the maximum deviation being less than 5.0 per cent on all asphalt except No. 9.

TABLE I.—COMPARATIVE RESULTS WITH DIFFERENT MAXIMUM TEMPERATURES OF RECOVERY.

Asphalt	Asphalt Tests	Original Asphalt	Asphalt Recovered by Bussow Method			
			225 C.[a]	250 C.[a]	275 C.[a]	300 C.[a]
No. 1—Texas	Penetration at 77 F. (25 C.)	49	59	53	51	49
	Loss at 325 F. (163 C.)	0.04%	0.33%	0.14%	0.16%	0.06%
	Penetration after heating	43	48	44	42	42
No. 2—Colombian	Penetration at 77 F. (25 C.)	64	65	64	64	62
	Loss at 325 F. (163 C.)	0.04%	0.28%	0.16%	0.07%	0.06%
	Penetration after heating	60	58	58	59	59
No. 3—Venezuelan	Penetration at 77 F. (25 C.)	88	92	86	85	84
	Loss at 325 F. (163 C.)	0.16%	0.45%	0.20%	0.18%	0.12%
	Penetration after heating	78	72	76	76	75
No. 4—Mexican	Penetration at 77 F. (25 C.)	97	103	100	97	98
	Loss at 325 F. (163 C.)	0.18%	0.45%	0.26%	0.20%	0.12%
	Penetration after heating	92	88	90	91	92
No. 5—Mexican-Colombian	Penetration at 77 F. (25 C.)	121	123	118	......	107
	Loss at 325 F. (163 C.)	0.10%	0.40%	0.20%	......	0.15%
	Penetration after heating	105	100	102	......	96
No. 6—Venezuelan	Penetration at 77 F. (25 C.)	201	205	204	176	190
	Loss at 325 F. (163 C.)	0.08%	0.29%	0.28%	0.20%	0.10%
	Penetration after heating	172	165	174	150	170
No. 7—Mexican	Penetration at 77 F. (25 C.)	205	215	200	197	200
	Loss at 325 F. (163 C.)	0.30%	0.35%	0.26%	0.32%	0.12%
	Penetration after heating	183	177	175	181	177
No. 8—Mexican	Penetration at 77 F. (25 C.)	235	243	229	222	......
	Loss at 325 F. (163 C.)	0.44%	1.00%	0.65%	0.50%	......
	Penetration after heating	200	192	195	189	......
No. 9—Mexican	Penetration at 77 F. (25 C.)	270	260	252	242	......
	Loss at 325 F. (163 C.)	0.21%	0.40%	0.15%	0.16%	......
	Penetration after heating	231	212	210	200	......

[a] Maximum temperature.

Comparison with Abson Method:

Fresh benzol solutions were made up of asphalts Nos. 4, 6, and 7, and the asphalt was recovered by Abson's method as well as the author's method. As Abson, in his paper, stated that he preferred a maximum temperature of 150 C. to the usual 163 C. for asphalts softer than 100 penetration, both temperatures were used for the Abson recoveries on asphalts Nos. 6 and 7.

The comparative results are given in Table II.

TABLE II.—COMPARATIVE RESULTS OF THE ABSON AND BUSSOW METHODS.

Asphalt	Asphalt Tests	Original Asphalt	Recovered Asphalt					
			Abson Method		Bussow Method			
			150 C.^{a}	163 C.^{a}	225 C.^{a}	250 C.^{a}	275 C.^{a}	300 C.^{a}
No. 4—Mexican...	Penetration at 77 F. (25 C.)	97	......	96	103	100	97	98
	Loss at 325 F. (163 C.)	0.18%	......	0.08%	0.45%	0.26%	0.20%	0.12%
	Penetration after heating	92	......	83	88	90	91	92
No. 6—Venezuelan.	Penetration at 77 F. (25 C.)	201	203	185	205-210	204-203	176-174	190-192
	Loss at 325 F. (163 C.)	0.08%	0.20%	0.04%	0.29-0.30%	0.28-0.30%	0.20-0.16%	0.10-0.12%
	Penetration after heating	172	161	155	165-170	174-172	150-147	170-171
No. 7—Mexican....	Penetration at 77 F. (25 C.)	205	300	240	215-226	195-200	197	200
	Loss at 325 F. (163 C.)	0.30%	0.64%	0.38%	0.35-0.32%	0.30-0.26%	0.32%	0.12%
	Penetration after heating	183	180	170	177-185	175-180	181	177

^{a} Maximum temperature.

In Table II, an unexpected result was obtained with the asphalts recovered by the Abson method. The penetration of the residue after the loss on heating test was always lower than on the residue from the loss on heating test on the original asphalt and was also lower than the penetration of most of the residues after loss on heating from the asphalts recovered by the Bussow method. This was true even when the loss on heating of the Abson recovered residue was lower than the loss on heating of the original asphalt. The number of Abson recovered asphalts reported is not sufficient to warrant drawing a definite conclusion as to the cause of the drop in penetration as compared with the results on the original asphalts.

Ductility of Recovered Asphalt:

Ductilities are not reported on any of the asphalts as they would all have been over 100 cm. In connection with ductilities of recovered asphalts, Steinbaugh and Brown[6] found that in the recovery of laboratory oxidized asphalts ranging from 17 to 53 in penetration and 38 to 80 cm. in ductility, there was an average loss of 2 points (6 per cent) in penetration and an average loss of 11 cm. (23 per cent) in ductility when recovered by the Abson method as compared with an average gain of 0.2 points (0.5 per cent) in penetration and an average gain of 20 cm. (40 per cent) in ductility when recovered by the Bussow method.

Steinbaugh and Brown concluded that the temperature of 300 C. used in the Bussow method was extreme when compared to the paving plant temperatures, which rarely exceed 190 C., and that the marked increase in ductility, more than 100 per cent in a few of their asphalts, was caused by the high temperature used in the recovery. They also concluded that the temperature of recovery was not a major factor in causing the loss of ductility in all the Abson residues.

The author cannot agree with their conclusions. In manufacturing blown asphalts at the refinery, temperatures in excess of 315 C. (600 F.) are used as compared to the 300 C. (570 F.) used in the author's method of recovering asphalts. The blown asphalts used by Steinbaugh and Brown were oxidized at temperatures of 480 to 500 F. (250 to 260 C.), considerably below the 570 F. (300 C.) temperature of the recovery. The author believes it to be necessary to heat the recovered asphalt to a temperature close to usual refinery temperatures in order to secure the same conditions of mutual solubility that control the ductility of the original asphalt. It is unfortunate that they did not heat their original oxidized asphalts to 300 C. or use a maximum recovery temperature of 260 C. and observe the effect on the ductility of the asphalts.

Steinbaugh and Brown found that all asphalts recovered by the Abson method when heated in a flask as in recovery by the Bussow method, gained in ductility and could be considered checks of the residues recovered by the latter method. This points to the obvious conclusion that the temperature of recovery is a very important factor in causing the loss in ductility of asphalts recovered by the Abson method.

The asphalt refiners learned long ago that when they altered the character of asphalts by blowing with air at relatively low temperatures, it was necessary to raise the temperature at the end of the blowing to insure the production of a good asphalt cement that would not become oily or waxy on standing. It seems logical to infer that where the character of an asphalt has been altered by dispersion in a solvent in which its component parts are not all equally soluble, the less soluble components will separate first upon the evaporation of the solvent, and may not be restored to their former condition of mutual solubility with the other components of the asphalt until the asphalt has been heated to the temperature at which it was originally formed, or to some lower temperature that will permit the same degree of mutual solubility of the component parts.

Conclusion:

Soft asphalts may be recovered by the Bussow method by using a maximum temperature of 250 C. but the author prefers a maximum temperature of 300 C. for asphalts less than 100 penetration. Asphalts up to 200 penetration may even be recovered at 300 C. as is shown by the loss on heating and penetration after heating of the residues recovered at that temperature. The only exception is asphalt No. 5 which was not a refinery product but was prepared in the laboratory by fluxing asphalts Nos. 2 and 8. This exception illustrates the weakness and danger of drawing conclusions from laboratory prepared samples as to the value and accuracy of asphalt recovery methods applied to commercially refined asphalts.

AN EXTRACTION METHOD FOR THE DETERMINATION OF METALS IN BOILED LINSEED OIL AND DRIERS

By Frank Gottsch[1] and Benjamin Grodman[1]

Synopsis

It has been suspected for a long time that the prevailing method employed to determine the amount of metals in boiled linseed oil and in driers was subject to considerable error. The impossibility of securing satisfactory duplicate results and the difficulty inherent in the operation of reducing the sample to ash for the analysis led to the development of the extraction method herein proposed in which the metals are extracted directly from a solution of the sample by means of suitable acids. The apparatus employed may be the usual laboratory equipment or a special type recommended in the A.S.T.M. Tentative Method of Test for Tetraethyl Lead in Gasoline (D 526 - 39 T).[2]

Description of the Ashing Method[3]

In the ashing method, 10 or 25 ml. of the sample are ignited by playing a flame of a burner over the surface. When most of the oil has quietly burned off, the crucible is reduced to ash in a muffle furnace or by direct heating at a dull red heat until all carbonaceous matter is consumed. The ash is dissolved in acids and the separation and determination of the metals are made in accordance with the A.S.T.M. Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (B 40 - 36 T).[4]

This method was employed for the estimation of lead, manganese, cobalt, and zinc in boiled linseed oil and driers. The results so obtained were found to be low and inaccurate in respect to lead and zinc, but fairly accurate in the case of cobalt and manganese.

At the best the ashing method requires careful manipulation and strict attention to the operation of ignition and ashing. With lead driers, a high ignition temperature leads to fusion of a part of the lead with the glaze of the porcelain crucible. The leaded glaze resists the action of the acid used in dissolving the ash, and thus some lead is lost. With zinc driers a high ignition temperature leads to loss by sublimation of a considerable portion of the total zinc content. With cobalt and manganese driers too high an ignition temperature leads to loss of their oxides by fusion with the glaze of the porcelain crucibles in case porcelain ware is used. This loss by fusion can be eliminated if platinum dishes are used.

Another difficulty encountered in the ashing method is that of loss by air drafts. If the ashing is not properly protected against drafts, a part of the ash may be blown away.

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[1] Principal Chemist, and Senior Chemist, respectively, Central Testing Laboratory, Department of Purchase, City of New York.

[2] 1939 Book of A.S.T.M. Standards, Part III, p. 630.

[3] Federal Specification JJJ-O-331; also Standard Methods of Testing Drying Oils (D 555 - 39), 1939 Book of A.S.T.M. Standards, Part II, p. 718.

[4] Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (B 40 - 36 T), 1939 A.S.T.M. Methods of Chemical Analysis of Metals, p. 91.

The Proposed Extraction Method

A method is proposed which eliminates the difficulties described for the ashing method, and which possesses the advantage that no special skill is required. It involves an acid extraction process whereby the metals are separated from their compounds in the form of their chlorides or nitrates. Holley[5] describes a method for determining the drying salts in Japans and driers based on the same principle of acid extraction, but his method differs from the proposed method in certain details. Bearn[6] suggests the acid extraction of lead from boiled linseed oil only as a qualitative test, but not as a quantitative one. The authors are not aware of any method for the analysis of metals in boiled linseed oil and driers employing the acid extraction principle in combination with the specific details further described.

Principles of the Extraction Method:

Dilution with benzol and boiling for 1 hr. complete the conversion and separation of the lead. The nitric acid and the hydrogen peroxide effect the oxidation of the oil. Without the nitric acid and the peroxide the oil is carbonized by the sulfuric acid but not completely destroyed. The carbonized oil later interferes greatly with the precipitation and filtration of the lead sulfate.

Apparatus:

Two types of apparatus were used in this investigation: (1) a 500-ml. round-bottom flask with short neck and ground-glass joint connected to an Allihn water condenser with bulbed inner tube, and electric heater with rheostat control, and a 300-ml. separatory funnel; (2) an electrically heated extraction apparatus accepted by the Society for the determination of tetraethyl lead in gasoline.[2]

The reagents employed are:

(*a*) Benzol (90 per cent grade).

(*b*) Dilute hydrochloric acid (one part acid to one part water).

(*c*) Concentrated nitric acid.

(*d*) Concentrated sulfuric acid.

(*e*) 3 per cent hydrogen peroxide.

Lead in Boiled Linseed Oil:

Using the first type of apparatus 34.17 g. of the oil (the equivalent of one half of the factor weight of lead in lead sulfate) are weighed and transferred into the 500-ml. flask, using benzol for washing. More benzol is added to make a total volume of 75 ml. benzol used. Then 75 ml. of dilute hydrochloric acid and a half dozen glass beads are added, and the flask is connected with the water condenser. The flask and contents are heated for 1 hr. at boiling temperature. Digestion for 1 hr. is adequate for the complete conversion of the lead compound into the water-soluble chloride.

The hot solution is transferred to the 300-ml. separatory funnel and the solution is allowed to settle for a few minutes or until the benzol layer separates out sharply and completely. The acid layer is removed and filtered through filter paper into a 400-ml. beaker. The benzol layer is transferred back into the flask, 75 ml. of water added and boiled as before for a few minutes. The hot solution is again transferred to the separatory funnel and the layers allowed to separate clearly. The aqueous layer is removed and filtered through the same paper into the beaker holding the first filtrate. To the filtrates, 5 ml. of concentrated nitric acid, 5 ml. of hydrogen peroxide, and 10 ml. of concentrated sulfuric acid are added. The determination of lead as lead sulfate is then made as usual.[7] Evaporation of the solution to SO_3 fumes should be carried out twice in order to insure the complete expulsion of the nitric acid. The weight of lead sulfate multiplied by two gives the percentage of metallic lead in the oil.

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[5] C. D. Holley, "Analysis of Paint and Varnish Products," 1912 Edition, John Wiley and Sons, Inc., New York, N. Y.

[6] J. Gauld Bearn, "Chemistry of Paints, Pigments and Varnishes," Ernest Benn, Limited, London, England (1925).

Inasmuch as a significant amount of siliceous matter has been found accompanying the lead sulfate, correction should be made by dissolving the lead sulfate as weighed with hot saturated ammonium acetate solution and washing the crucible with hot water until free from salts. The weight of the ignited residue is deducted from the total. The siliceous matter originates from the action of the acids and the peroxide upon the glass beaker.

Using the second type of apparatus the operations of transferring and retransferring the solutions to a separatory funnel are eliminated, resulting in the saving of time and labor.

The same quantity of sample as was used for the first type of apparatus is taken and, as before, transferred by means of benzol to the special apparatus described in D 526.[2] Seventy-five milliliters of dilute hydrochloric acid are added, and heated to gentle boiling by the electric coil heater for 1 hr., the heat being controlled by means of the rheostat. The heat is discontinued and the contents allowed to settle for a minute or two. The lower acid layer is then filtered into a 400-ml. beaker. Seventy-five milliliters of water are added to the extractor, boiled for a few minutes, and filtered through the paper into the beaker containing the first filtrate. The procedure is then as described for the first type of apparatus.

Lead in Driers:

The procedure for the determination of lead in driers is the same as for linseed oil in all but two respects, namely, the size of the sample and the kind of acid used for the extraction. The size of sample depends upon the metallic lead content. For a drier containing 32 per cent of metallic lead 3 to 5 g. are ample; for a drier of a much lower metal content a larger sample should be taken. Dilute nitric acid (1:2) is used instead of dilute hydrochloric acid in view of the greater solubility of the lead nitrate in water. This allows of the taking of an aliquot portion for the lead determination without the trouble caused by the separation of the lead salts. In case calcium is also present in the drier, the lead is first separated from the calcium by hydrogen sulfide, and then precipitated as the sulfate.

Manganese in Driers:

In determining manganese in driers, the extraction is carried out as described for lead in driers. The extracted solution is transferred to a 200-ml. volumetric flask and made up to volume. A 25-ml. aliquot is removed into a 250-ml. beaker or Erlenmeyer flask, 25 ml. of dilute nitric acid (1:1) added, and boiled for 10 min. Sodium bismuthate is added sufficient to precipitate only part of the manganese. The boiling, is continued for a few minutes, and then the manganese oxide is reduced with a minimum amount of sulfurous acid or ferrous sulfate.[8] After boiling the solution is cooled in cold water. An excess of sodium bismuthate is added to insure the complete oxidation of the manganese to the permanganate state. The solution is filtered through an asbestos pad to remove the excess bismuthate, and then titrated with standard 0.1 N ferrous ammonium sulfate. In the presence of cobalt the manganese should be determined by the ammonium persulfate and arsenite method, but not by the bismuthate.[9]

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[7] Standard Methods of Chemical Analysis of White Pigments (D 34 - 39), 1939 Book of A.S.T.M. Standards, Part II, p. 676. (Lead as sulfate.)

[8] Standard Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron and Wrought Iron (E 30 - 39), 1939 A.S.T.M. Methods of Chemical Analysis of Metals, p. 1.

TABLE I.—PERCENTAGES OF METALS FOUND IN BOILED LINSEED OIL AND DRIERS.

Material	Lead		Manganese		Cobalt		Zinc	
	Ash Method	Extraction Method	Ash Method	Extraction Method	Ash Method and Deposition	Extraction Method and Deposition	Ash Method	Extraction Method
Boiled Linseed Oil No. 1......	0.0158 0.0183	0.0194 0.0196						
Boiled Linseed Oil No. 2......	0.0178 0.0160	0.0220 0.0228						
Boiled Linseed Oil No. 3......	0.0120	0.0180 0.0196						
UVSL Liquid 32% No. 1......	29.71 30.01 30.11	32.40 32.39 32.41						
UVSL Liquid 32% No. 2.......	31.38 32.72	32.41 32.36						
UVSL Liquid 6%.............	......	......	5.80 5.98	5.88 5.90				
UVSL Liquid 6% No. 1........	......	......	....	....	6.12 6.17	6.41 6.43		
UVSL Liquid 6% No. 2........	......	......	....	....	6.20 6.32	6.34 6.42 6.40		
UVSL Liquid 8%.............	......	......	....	....	....	....	6.97 6.45 6.52	7.83 7.89 7.88
NUDX 24% No. 1............	23.0	23.68 23.62						
NUDX 24% No. 2............	23.13	23.49 23.51						
NUDX 6%..................	......	......	5.68 5.80	5.74 5.78				
NUDX 8%..................	......	......	....	....	....	....	6.92 7.20	7.63 7.70

Cobalt in Driers:

Except for the acid the procedure for extracting the cobalt from driers is the same as for lead or manganese in driers. Dilute hydrochloric acid (1:1) is used instead of nitric. The size of the sample taken for analysis depends upon the metal content. A convenient and satisfactory method for determining the metal is the electrolytic one described by Hillebrand and Lundell.[10] Either all or an aliquot portion of the extracted solution is used for the analysis. In case lead, manganese, etc., are also present in the sample, the cobalt should be separated from them by a standard procedure before pursuing with the electrolysis. The electrolytic method in brief is as follows:

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[9] H. A. Gardner, "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," Ninth Edition, p. 307, Institute of Paint and Varnish Research, Washington, D. C. (1939).

[10] W. F. Hillebrand and G. E. F. Lundell, "Applied Inorganic Analysis," 1929 Edition, John Wiley and Sons, Inc., New York, N. Y.

To the cobalt chloride solution in a 250-ml. beaker add ammonium hydroxide until slightly alkaline. Filter if necessary. Add 20 g. of ammonium sulfate, 2 g. of sodium bisulfite, and 35 ml. of concentrated ammonium hydroxide. Dilute to about 200 ml. and electrolyze, using platinum gauze electrodes and a current density of 0.2 to 0.3 amp. per sq. dm. The electrolysis is complete when the solution turns colorless. Wash the deposit with water and alcohol, and dry at 100 C. for a few minutes. The cobalt deposit should be free from black stains. Black stains indicate oxidation, and the deposit calculated as pure cobalt is incorrect. The black stains can be prevented by keeping the gauze electrode totally immersed in the liquid during the electrolysis.

Zinc in Driers:

In determining zinc in driers, the extraction is carried out as described for cobalt in driers. The extracted zinc chloride solution is received into a 400-ml. beaker and boiled for several minutes to expel the small amount of benzol. The excess acid is neutralized with ammonium hydroxide, 6 ml. concentrated hydrochloric acid added, and the solution diluted to 300 ml. It is heated to boiling and titrated with standard potassium ferrocyanide solution.[11]

SUMMARY AND CONCLUSION

The metals lead, manganese, cobalt and zinc are extracted completely from their organic compounds contained in boiled linseed oil and driers by use of either dilute hydrochloric or dilute nitric acid with the assistance of heat and benzol.

Table I shows the results obtained by the ashing method and by the extraction method for lead, manganese, cobalt and zinc in boiled linseed oil and naphthenate driers. These results show that the extraction method as proposed is accurate and dependable for all of the four metals tested, while the ashing method is satisfactory for only manganese and cobalt.

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[11] Tentative Methods of Chemical Analysis of Silver Solders (B 81 - 36 T), 1939 A.S.T.M. Methods of Chemical Analysis of Metals, p. 189.

DISCUSSION

Mr. H. E. Smith.[1]—A good many years ago I tried the cold extraction of lead from boiled linseed oil dissolved in ether, but found it was a difficult subject. After 10 or 15 washings with dilute nitric acid there was still a little lead left in the oil. The direct ignition of organic matter containing lead or zinc always results in loss. To overcome this, in determining the driers in either oil or japan driers, I have used a modified method of ashing the organic matter. Instead of burning the sample directly, the dish is set high over a lamp and the oil evaporated without ignition. If it happens to take fire, the dish is covered immediately and after it has cooled somewhat, the evaporation is started again. In this way the oil can be brought to a char at a very low temperature. The char is extracted with hot dilute nitric acid, filtered and washed, after which the char burns readily. If only the "total driers" is wanted, next add the nitric acid solution to the ash in the dish, evaporate and ignite. If the drier metals are to be determined separately, it is better not to ignite, but to dissolve the ash in hot hydrochloric acid and combine the solution with the nitric acid extract.

For the final determination of manganese, I prefer to titrate with arsenite or thiosulfate rather than to use ferrous sulfate.

Recently it appears that naphthenate driers are somewhat volatile, therefore if they are present, even the evaporation of the oil may result in some loss. The method now proposed by the authors presumably overcomes this difficulty.

Mr. A. E. R. Westman.[2]—I should like to ask the author whether this method would have any application to dried films, such as varnish scrapings.

Mr. Frank Gottsch.[3]—In reply to Mr. Westman—I think it would not because the dry films would be very difficult to dissolve. The method might be modified. The essential feature of this method is benzol, the universal organic solvent for these materials, and extraction of the solution by nitric acid or hydrochloric acid. If you can get the films dissolved in some suitable organic liquid, then you could proceed; or you might dissolve them first in some way in acids, and then further, use an organic solvent. You would have to work that out.

Mr. H. Levin.[4]—I should like to draw attention to a somewhat similar method[5] employed in the petroleum industry for determining lead soap in car oil. Benzene (benzol) is employed as solvent. The method employs benzene and glacial acetic acid for decomposition, thereby having a single phase instead of two phases. We confirm the authors' experience of the convenience of this apparatus for decomposition. I do not mean that we use it primarily in the determination of lead soap, but we use it for other purposes and it is a very convenient apparatus.

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[1] Materials Engineer, White Plains, N. Y.

[2] Director of Chemical Research, Ontario Research Foundation, Toronto, Ont., Canada.

[3] Principal Chemist, Central Testing Laboratory, Department of Purchase, City of New York.

[4] Supervisor, Analytical and Testing Dept., The Texas Company, Beacon, N. Y.

[5] Levin, *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 6, p. 333 (1934).

MR. GOTTSCH.—I understand that Mr. Levin uses the apparatus for lead in gasoline.

MR. LEVIN.—That is right.

MESSRS. FRANK GOTTSCH and BENJAMIN GRODMAN[6] (*Authors' closure by letter*).—The usefulness of this method for the examination of dried films of varnish has been suggested by the comments. The dried films can be dissolved in a regular type of varnish remover and procedure given can then be followed for the determination of the various metallic driers. A commendable cooperation now exists between the American Society for Testing Materials and various Governmental laboratories and departments in extending knowledge of driers. It is hoped that such joint efforts may be broadened to include many manufacturers and consumers by the promulgation of similar research publications, leading to the formulation of intelligent specifications for this kind of material.

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[6] Senior Chemist, Central Testing Laboratory, Department of Purchase, City of New York.

A CORRELATION OF THE CHEMICAL AND ELECTRICAL CHANGES OBSERVED DURING THE OXIDATION OF MINERAL INSULATING OIL

By F. M. Clark[1]

Synopsis

There is no single test which can be used as a criterion of the chemical and dielectric quality and condition of mineral transformer oil for commercial use. The effect of oil oxidation on the chemical and electrical properties of mineral transformer oil and their relation to each other is determined by the nature of the crude stock from which the transformer oil is prepared, the type and degree of the refining treatment used in the manufacture of the finished product, and the conditions applying at the time of the oxidation. A variety of chemical and electrical tests give information concerning the nature and amount of change occurring during the oxidation. Each test is significant within the limits of its own scope, but in a practical sense each gives only general information concerning the probable over-all condition of the oil. Of those tests which are of practical utility in the study of oil in commercial use, the color and acidity values appear of some importance although laboratory investigations have shown that the color and acidity change in mineral transformer oil cannot be used as a final gage of the potential sludge formation characteristics. With continued oxidation a condition is eventually reached where the sludge-forming propensity of the oil increases rapidly with but little further increase in the color or acidity value. Study of the changes in the color and acidity of oil in individual transformers in commercial use, however, with frequent reference to a laboratory test for evaluating the potential sludge-forming qualities of the oil should protect against the continuation in service of a chemically unstable material.

The dielectric strength test has but little direct relation to the chemical changes occurring in mineral transformer oil during oxidation except in so far as it is affected by the increase of water solubility in oxidized oil. The dielectric strength of the oil is, therefore, best controlled by its own specific test result. The power factor of the oil must be considered as a dielectric test distinct in its behavior from the dielectric strength and worthy of general practical consideration in proportion to the importance of dielectric loss in the successful operation of the transformer.

Mineral oil has been exhaustively studied by numerous investigators in attempts to evaluate the chemical and electrical changes which occur during its oxidation. The object of these studies has been to provide a reliable test for the commercial selection of oils of high resistance to chemical and electrical deterioration. A second object has been to provide a means for determining when a deteriorated oil can no longer be safely continued in commercial use. A variety of tests have been suggested. Among these have been the physical and chemical tests for viscosity, color, refractive index, specific gravity, total and water-soluble acidity, hydrophil number, saponification, and sludge formation. The electrical tests suggested include the dielectric strength, resistivity, power factor and dielectric constant. Generalizations based on these or other special types of tests as affected by the oxidation of the oil during an empirically set up laboratory oxidation have been frequently found to be misleading and often in direct conflict. It is an object of the present paper to study the possible correlation existing between the more important oil properties in order that attention may be closely focused on those which appear worthy of further study.

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In normal commercial practice, two oil properties have been generally recognized as being of major importance for the successful use of mineral transformer oil. These have been the ability of the oil to retain a high dielectric strength and to resist those factors which promote sludge formation as a result of oxidation. The dielectric strength test on mineral transformer oils is in wide commercial use, but despite the importance of the sludging factor, no general agreement has been reached concerning the method best adapted for estimating the sludge-forming properties of the oil. From a practical standpoint, the increase in oil color and in oil acidity have been widely used to protect against undue oil oxidation leading to dangerous sludge deposition during the use of the oil in transformers. More recently the power factor test has been advocated as of fundamental importance in indicating the general chemical and dielectric condition of the oil in service.

The difficulty underlying the problem of transformer oil testing is largely due to the fact that the properties of major importance for the successful functioning of the oil in service are both chemical and electrical. To date no one property has appeared to encompass the whole range of requirements so that by its study the chemical and electrical condition of the oil can be safely guaranteed. The problem is made still more difficult by the fact that many oil-soluble contaminants are known which affect one type of oil property, chemical or electrical, without producing more than a negligible effect on the other. These difficulties have led many operators of mineral-oil-filled transformers to place little if any reliance on the laboratory examination of oil drawn from transformers in commercial service. Instead, periodical examination of the transformer core and coils is made and the presence or absence of insoluble sludge is noted. A continuation of this practice, together with the careful analysis of the oil drawn from the transformer at regular intervals during operation should eventually enable the laboratory technician to establish a satisfactory method for gaging the suitability of the oil for continued use.

In the following analysis, attention is directed to a consideration of the following tests, the methods for determining the more important of which are described in the Appendix,[2] following as far as possible the published procedures sponsored by the American Society for Testing Materials:

Oil color
Total acidity
Water-soluble acidity
Viscosity
Potential sludge formation
Refractive index
Specific gravity
Saponification number
Ester value
Hydrophil number
Dielectric constant
Resistivity
Power factor
Dielectric strength

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[2] See p. 1234.

Of these tests, several have been shown to have but little value in the study of oil deterioration, and, for reasons of brevity, are eliminated from further consideration. These tests include those for viscosity, refractive index, specific gravity, and dielectric constant.

It must be clearly understood that the only factor considered in this paper is the relation existing between two or more properties of the oil as affected by oxidation and use. The time factor is eliminated from consideration. The fact that an oil may show, under certain conditions of oxidation, a rapid increase in the potential sludge formation value at a definite oil acidity does not necessarily indicate that the oil has but little oxidation and sludge resistance. Indeed it may be of high resistance to oxidation and sludge formation and may approach the critical acidity value only under extremely severe oxidation treatment. Numerical limitations for allowable oil deterioration in commercial use, as is subsequently pointed out in the discussion, are difficult to set up. The chemical conditions applying during the oxidation of the oil greatly affect the type of oxidation and the products formed.

The purpose of the present paper will be attained if the result is the stimulation of further study in the correlation of those chemical and physical changes which occur during the oxidation of mineral transformer oil in the laboratory and in the field. Knowledge concerning the relation existing between such correlations and the practical oil experience as revealed by periodic examination of the transformer during use is a necessary prerequisite to the establishment of a laboratory test for gaging the commercial suitability of new and used mineral transformer oils.

The Hydrophil Test:

A general inspection of the hydrophil test results reveals many interesting correlations. In general, however, the hydrophil value varies as a linear function of the total oil acidity. The relation established is illustrated in Fig. 1. Since the hydrophil test appears to reflect those factors generally expressed in terms of total oil acidity, the hydrophil test is eliminated from further consideration in favor of the more commonly used test for the acidity value.

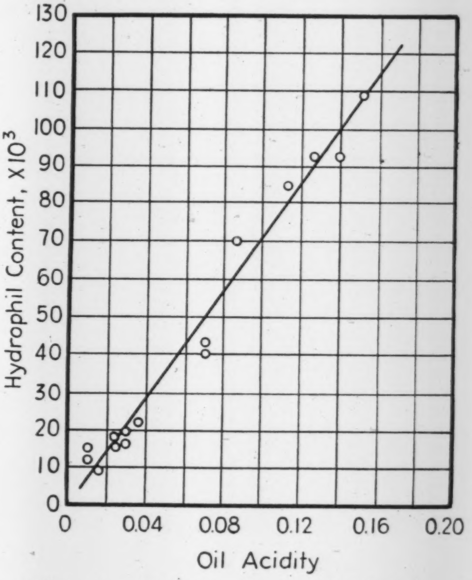

FIG. 1.—Relation Between the Acidity of Mineral Transformer Oil and Its Hydrophil Content.

The Resistivity Test:

The oil resistivity and power factor test values measured at 100 C. have yielded the relation indicated in Fig. 2. For oil resistivities lower than approximately 5 × 10^{12} at 100 C., the power factor changes more rapidly with further oil deterioration than does the resistivity and is considered as of greater value in the study of oxidized oils. The

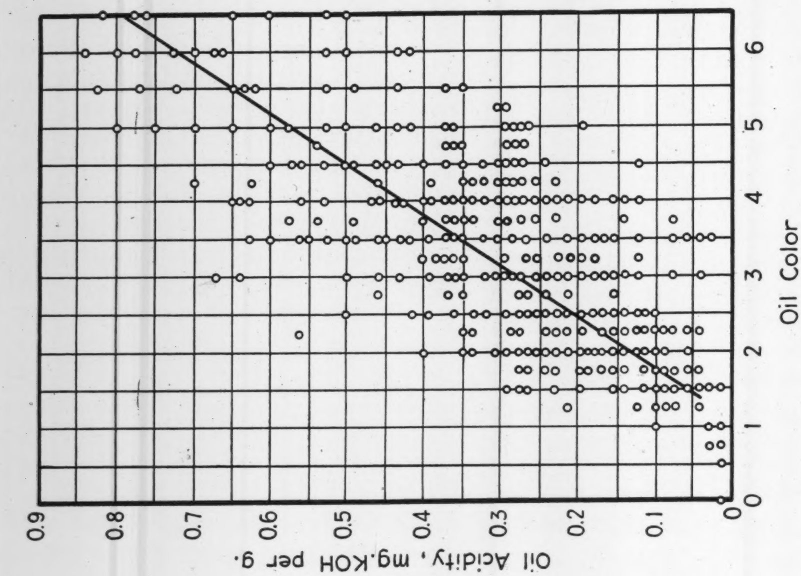

FIG. 3.—Relation Between the Color and Acidity of Mineral Transformer Oil Drawn from Commercially Operating Transformers.

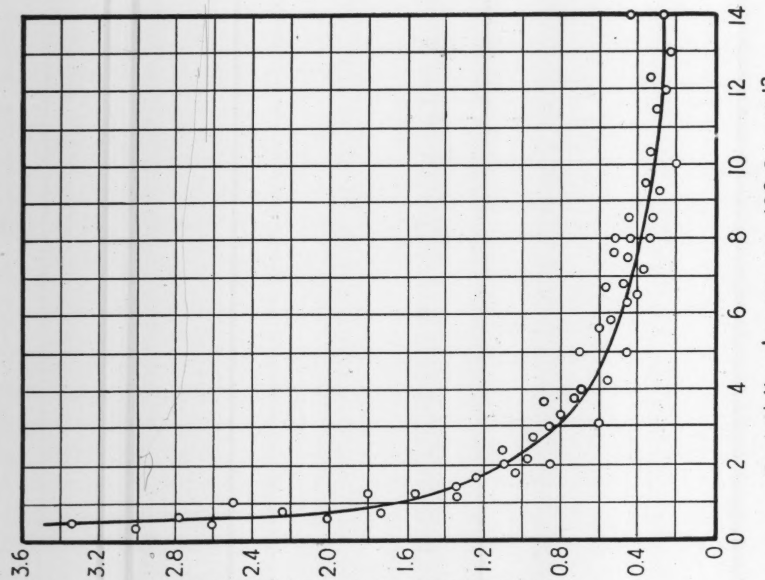

FIG. 2.—Relation Which Exists Between the 60-cycle Power Factor and the d-c. Resistivity of Mineral Transformer Oil as Affected by Oxidation.

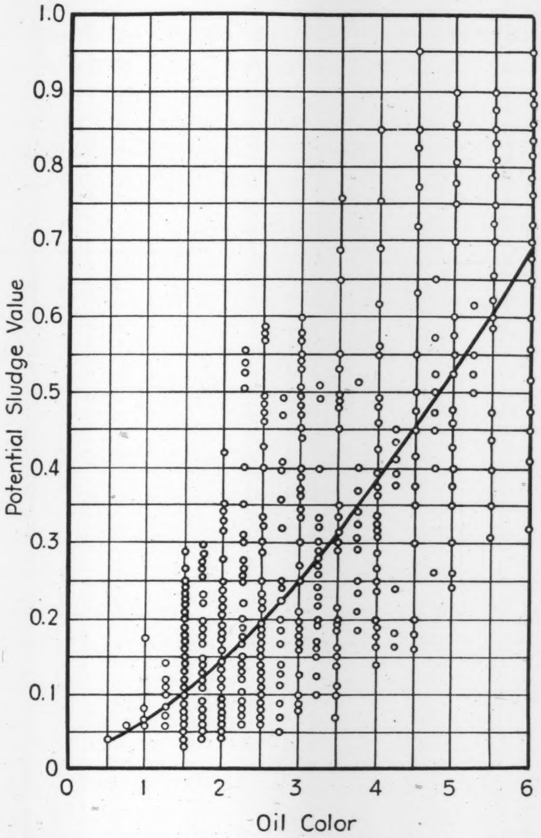

FIG. 4.—Relation Between the Color and the Potential Sludge Value of Mineral Transformer Oil Drawn from Commercially Operating Transformers.

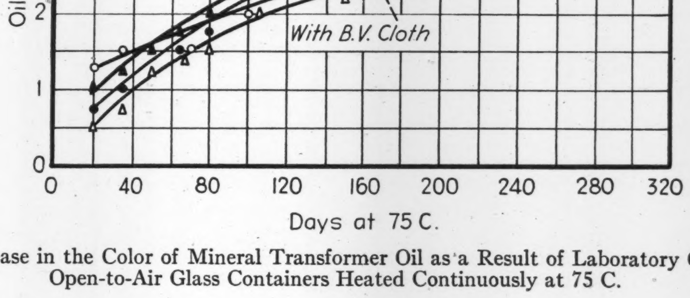

FIG. 5.—Increase in the Color of Mineral Transformer Oil as a Result of Laboratory Oxidation in Open-to-Air Glass Containers Heated Continuously at 75 C.

power factor has, therefore, been selected for detailed examination in preference to the oil-resistivity value.

The Color Test:

The oil color test has been widely used as a means for studying the stability of new oils and the degree of oil deterioration during transformer use. Since oil color in the commercial sense is frequently determined by the presence of oil-soluble materials which may have little effect on the important property of sludge formation, the use of oil color as a gage of oil condition in service is at once open to serious criticism. Figure 3 relates the oil color to the measured acidity for oil drawn from commercially operating transformers. Figure 4 relates the oil color to the potential sludge-forming property of these oils as evidenced by the high pressure oxidation test. In each instance, although there is a general relationship between the oil properties of color, acidity and of potential sludge formation, the wide extremes of the relation established clearly indicate the limitations of the color value as a criterion of oil quality.

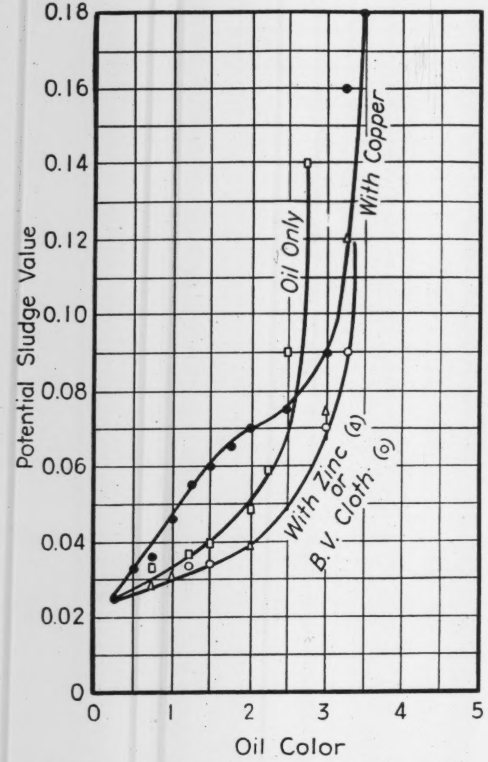

FIG. 6.—Despite the Apparent Stabilization in the Color of Mineral Transformer Oil After Approximately 200 days of Laboratory Oxidation at 75 C. (as Indicated in Fig. 5), the Potential Sludge Value Continues to Increase Indefinitely.

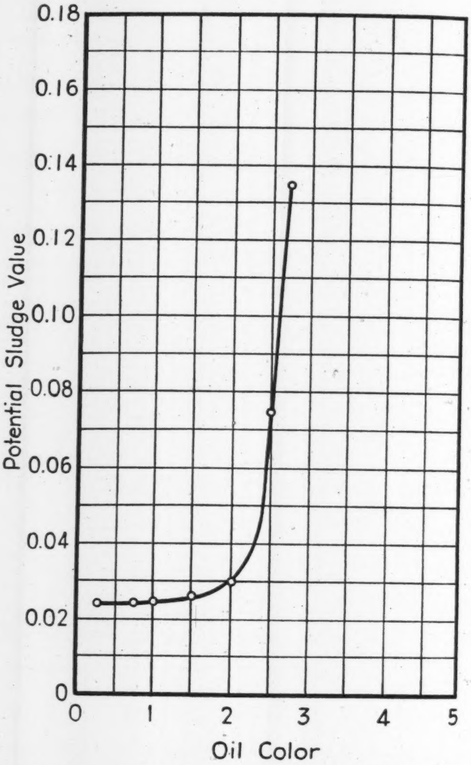

FIG. 7.—Indefinite Increase in the Potential Sludge Value of Mineral Transformer Oil at a Substantially Constant Color Value Attained After Approximately 200 days of Laboratory Oxidation in Open-to-Air Glass Containers Heated at 75 C. (as Illustrated in Fig. 6) also Occurs when the Oil Is Oxidized in Sealed Glass Vessels Containing 15 per cent Air.

From a strictly laboratory standpoint, however, the color test yields interesting information with respect to the potential sludge-forming properties of the oil. Mineral transformer oil has been oxidized at 75 C. in glass containers open to free contact with the unconditioned air of the heating oven. The temperature of 75 C. was selected as falling within the normal operating range of temperature to which the oils of Figs. 3 and 4 have been subjected. The oxidation test was continued for approximately 300 days. The oil color increased as is illustrated in Fig. 5. After approximately 200 days on test, the oil color appears to have reached a stable maximum value. But the deterioration of the oil has not ceased. This is clearly indicated in the data of Fig. 6 which relate the potential sludge-forming properties of the oil as a function of the oil color. Whether the oil be oxidized alone or in the presence of zinc, copper, or of black varnished cloth, the sludge-forming tendency of the oil oxidized at 75 C. increases independent of any substantial change in oil color when the color value has increased to the range of $2\frac{1}{2}$ to $3\frac{1}{2}$, A.S.T.M. As indicated in Fig. 7 the same lack of relation between oil color and potential sludge formation is evident also when the oil is oxidized at 75 C. in sealed containers, filled to 85 per cent of their

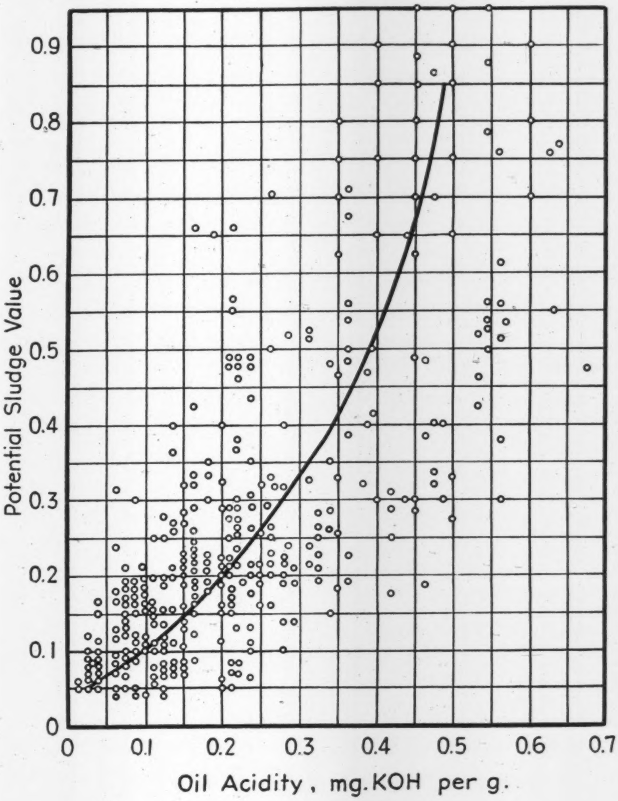

FIG. 8.—Relation Between the Potential Sludge Value and the Acidity of Mineral Transformer Oil Drawn from Commercially Operating Transformers.

volume with transformer oil, the remaining volume being filled with unconditioned air of 30 per cent relative humidity at 25 C.

As a practical guide to the operator of mineral-oil-filled transformers, the oil-color test may be of value in that any rapid change in the color of an oil in any particular installation is clearly worthy of a more detailed investigation. As a criterion of oil quality in a general and fundamental sense, however, the oil color test has little if any real value.

The Acidity Test:

A property in wide practical use, second only to that of color is oil acidity. Invariably oil acid values are accepted by the practical transformer engineer as furnishing a real clue to oil condition. Oil acidity does increase with the increase in oil color during service, but the wide variation in the relation shown to exist between these properties (Fig. 3) clearly indicates that as a criterion of oil condition, the acidity determination is of doubtful value. This conclusion is supported by the data of Fig. 8 which illustrate the relation obtained between the potential sludge formation and the total oil acidity. The data of Fig. 8 are based on tests made on oil drawn from transformers in commercial operation. In general an increasing oil acid value is accompanied by an increased potential sludging value but the wide variation in the potential sludging value for a fixed acidity as obtained from samples of oils drawn from a variety of different transformers indicates that in a strict sense no fixed acid value can be set up as a limit for normal safe operation, although a carefully preserved oil acidity record accumulated during commercial use in a particular oil-filled transformer may be of assistance in securing satisfactory oil service.

An inspection of Fig. 8, describing the total data obtained on oils drawn from numerous transformers in commercial use, reveals a tendency for the potential sludge value of the oil to increase without further increase in oil acidity above an acidity value of approximately 0.55. This limiting value of acidity, however, must be considered only as a guide in the most general sense. The exact relation between the potential sludge value and the oil acidity depends to a large extent on the condition of the oxidation, the type of transformer, the materials used in the transformer construction, the temperature conditions applying and other factors which influence the rate of oil oxidation and the type of oxidation products formed. This is indicated by the data illustrated in Figs. 9 and 10.

Figure 9 shows the relation of oil acidity and potential sludge formation in distribution transformers operated with a continuously applied load giving a constant oil temperature of 80 C. It will be observed that in accordance with the data of Fig. 8, the potential sludge value increases with increased oil acidity. In confirmation of Fig. 8, as the oxidation progresses, there is ultimately reached a condition in which the potential sludge value of the oil increases without further substantial increase in the acidity value. Unlike the data of Fig. 8, however, this "runaway" condition occurs at a much higher acid value. In the transformers of Fig. 9, the indefinite increase in potential sludge value without material change in the acidity occurred in the range of about 3.0 mg. of KOH per gram of oil.

Figure 10 illustrates the results obtained in a typical laboratory oxidation run. In order to avoid the criticism of the use of abnormally high oxidation temperatures, a criticism which is usually raised against laboratory investigations, the data of Fig. 10 were obtained during

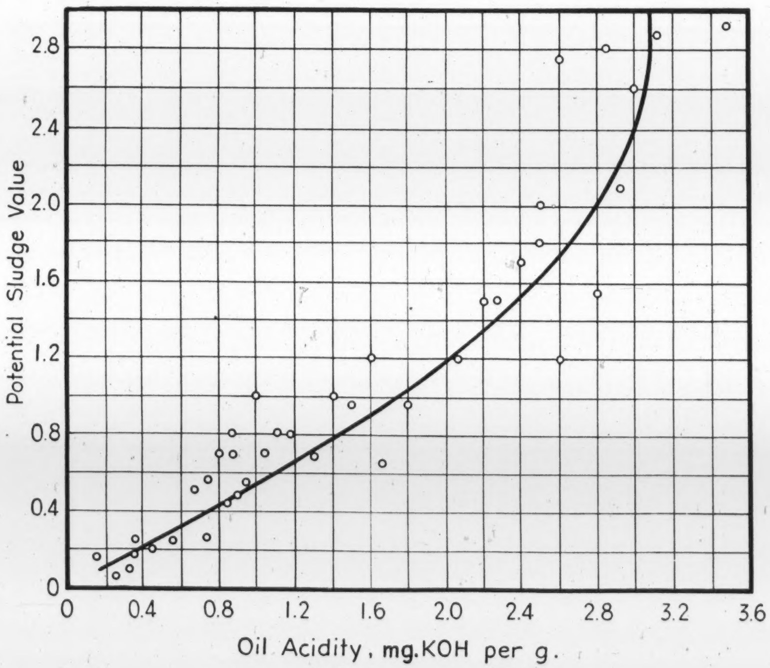

FIG. 9.—Relation Between the Potential Sludge Value and the Acidity of Mineral Transformer Oil Drawn from Transformers Operating with a Continuous Oil Temperature of 80 C.

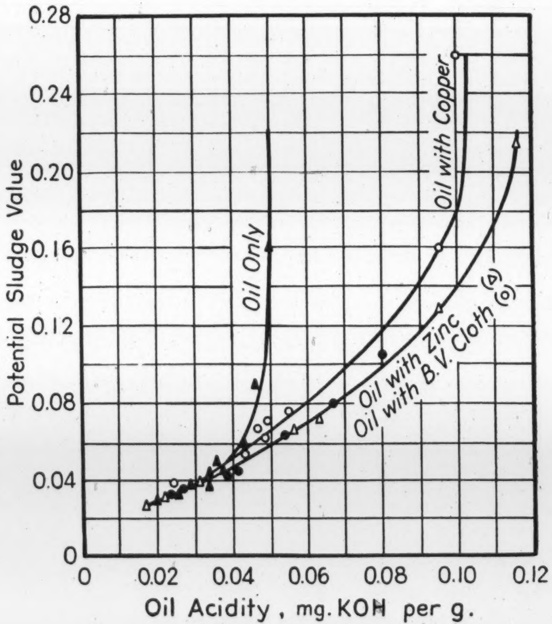

FIG. 10.—Under the Conditions of Laboratory Oxidation in Open-to-Air Glass Containers Heated Continuously at 75 C., the Potential Sludge Value of Mineral Transformer Oil Increases with the Increase in Oil Acidity to an Acid Value Beyond Which the Potential Sludge Value of the Oil Increases Rapidly with but Little Further Change in the Oil Acidity.

The oil acidity at which this runaway condition occurs is dependent on the conditions of the oxidation.

an oxidation run at 75 C. in open glass containers. The data are of interest and importance in indicating that the exact value of oil acidity beyond which there occurs a "runaway" in the potential sludge-formation value is extremely variable and entirely dependent on the conditions of the oxidation. With oil alone, under the conditions of the laboratory set-up, the potential sludge-formation value increases indefinitely without further increase in oil acidity at an acidity value of approximately 0.05 mg. of KOH per gram. Oxidized in the presence of copper, or in the presence of metallic zinc or black varnished cloth, the "runaway" condition occurs only at materially higher values. With copper, the value is in the range of 0.10 mg. of KOH per gram. In the presence of zinc or black varnished cloth, the "runaway" value occurs at an acidity of approximately 0.12 mg. of KOH per gram of oil.

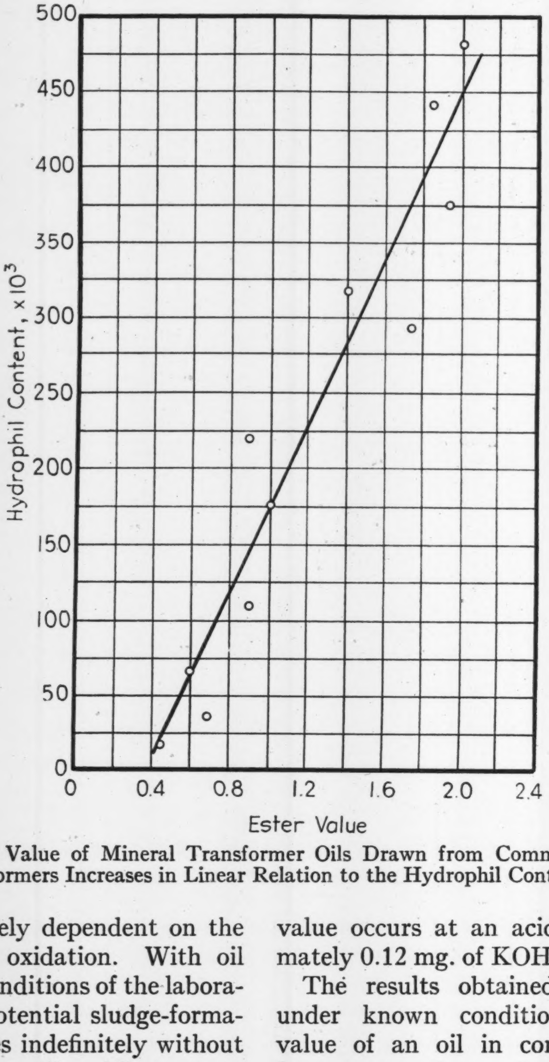

FIG. 11.—The Ester Value of Mineral Transformer Oils Drawn from Commercially Operating Transformers Increases in Linear Relation to the Hydrophil Content.

The results obtained indicate that under known conditions the acidity value of an oil in commercial transformers may be used to give valuable information concerning the suitability of the oil for further commercial use. Except in a very general sense, however,

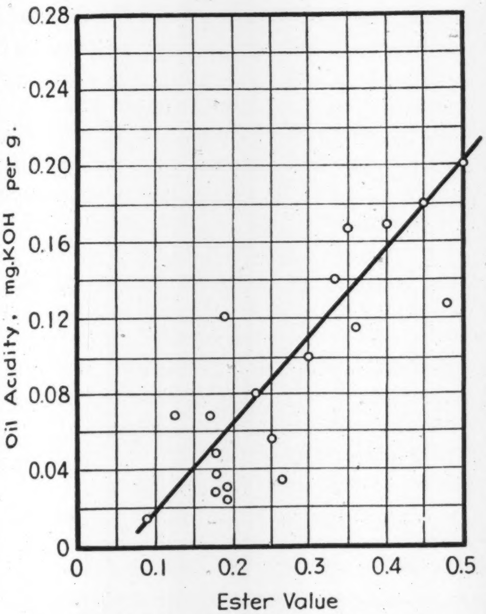

FIG. 12.—A Linear Relation Exists Between the Ester Value and the Acidity of Mineral Transformer Oils Drawn from Commercially Operating Transformers.

the acidity value cannot be safely used as a final criterion of oil quality.

The Ester Value:

The ester value is defined as the saponification number minus the total acidity value. The relation of the ester to other oil properties as affected by oil oxidation bears strong resemblance to the total acidity value. Figure 11 illustrates the relation between the ester value and the hydrophil number of commercially used oils. Figure 12 gives the corresponding relation between the ester value and the total acidity value. A linear relation is obtained between the ester value and the hydrophil and total acidity value of the oil as affected by commercial use.

It has already been illustrated (Fig. 10) that under laboratory oxidation conditions, transformer oil increases in its potential sludge-forming properties as the oil acidity increases until with con-

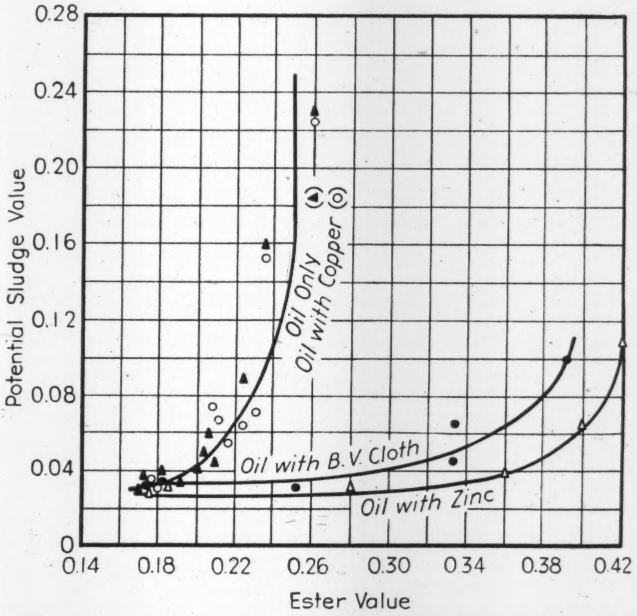

FIG. 13.—When Oxidized at 75 C. in Open-to-Air Glass Containers Under Laboratory Conditions, the Potential Sludge Value of Mineral Transformer Oil Increases with the Ester Value to a Point Beyond Which the Potential Sludge Value Increases Indefinitely Without Further Substantial Increase in Ester Content.

The "stable" ester value at which this runaway condition of the potential sludge value occurs is dependent on the conditions of the oxidation.

tinued oil oxidation, a condition is obtained at which the potential sludge value increases in a "runaway" manner without further increase in oil acidity. A similar result is obtained when the potential sludge-forming characteristic of the oil is related to the ester value. This is illustrated in Fig. 13. With continued oxidation, potential sludge-formation increases with the ester content to a point beyond which the sludge characteristic increases without further substantial change in the ester value. The ester content, however, like the property of total acidity already described, has no fixed limitation of fundamental significance since the "runaway" sludge condition is obtained at different ester values, dependent on the conditions under which the oxidation of the oil is caused to occur. In the presence of zinc or of black varnished cloth, the "runaway" character of the sludging characteristic is obtained at higher ester values. The only outstanding difference shown by the ester value as compared to the total acidity of the oil in its relation to the potential sludge value is to be observed when the oxidation is carried out in the presence of copper. Tested alone or in the presence of copper, the potential sludge-formation value of the oil, reaches a "runaway" condition at approximately the same ester content. In the case of total acidity, the "runaway" sludging characteristic is observed at a higher acid value when copper is present (Fig. 10).

Although the ester value may be worthy of further study as a criterion of oil quality, its marked resemblance to the more commonly used total acidity number justifies its elimination from further consideration in the present analysis.

The Potential Sludge-Formation Value:

The sludge-forming characteristic of a mineral transformer oil is usually determined in the laboratory by an empirically set-up test which gives accelerated oxidation. In discussing the results obtained by such methods, it must be clearly understood that when an oil is so tested in the laboratory, its *actual* sludging property under the conditions of commercial use is not determined. The only thing which is determined is the summation of those changes in the oil which reduce its resistance to oxidation and sludge formation under the artificial laboratory conditions set up. Since the oxidation and sludge-forming reactions in mineral transformer oil are very markedly affected by the unavoidable laboratory exaggeration of the testing conditions, the results obtained from such tests are designated in this paper as "potential sludge-formation values." The increase in these values indicates a decrease in the resistance of the oil to oxidation and sludge formation. Whether these changes are of commercial importance depends very largely on the operating temperature of the transformer. In a transformer, the oil temperature of which is maintained at lower than 50 C., for instance, danger from sludge formation in use would be but little increased by a relatively large change in the potential sludge-formation value. As the conditions of transformer operation become more severe from the standpoint of oil oxidation, an increase in the potential sludge-formation value of the oil becomes more important. It is the lack of uniformity in the possibility of oil oxidation in commercial use necessarily resulting from differences in transformer design and operation which prevents broad generalizations in this important field of oil technology.

The potential sludge-formation value of a transformer oil is estimated in this study by the amount of sludge formed in the oil as a result of a 24-hr. oxidation at 140 C. under a pressure of 250 lb. of oxygen. This test has been described in a previous publication[3] and has the advantage of being of short duration. It is operated under conditions such that substantially no oil evaporation or volatilization of oxidation-formed products occurs during the oxidation run. The potential sludge-formation value does not include consideration of the sludge actually precipitated from the oil as a result of previous oxidation or use.

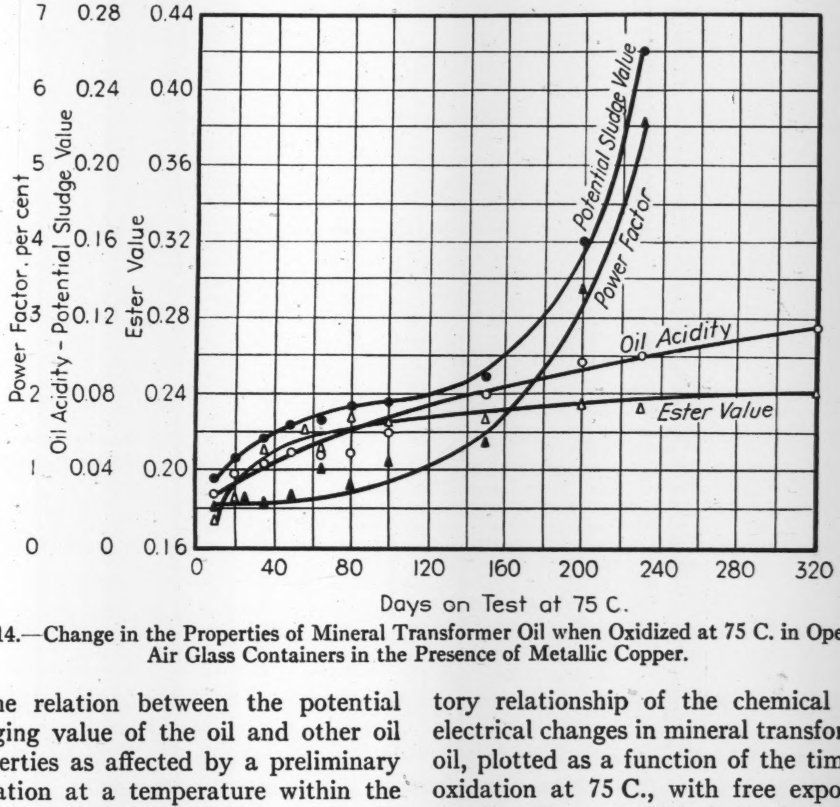

FIG. 14.—Change in the Properties of Mineral Transformer Oil when Oxidized at 75 C. in Open-to-Air Glass Containers in the Presence of Metallic Copper.

The relation between the potential sludging value of the oil and other oil properties as affected by a preliminary oxidation at a temperature within the normal operating range of transformers has already been illustrated in Figs. 4, 6, 7, 8, 9, 10 and 13. Whether considered in relation to the changing values of color, acidity, or ester produced by low temperature oxidation, the potential sludge-formation characteristic increases during the early stages of the oxidation with the increase in the second oil property considered. Eventually, with continued oil oxidation, a point is reached beyond which the potential sludge-formation value increases rapidly with but little further increase in the second oil property (color, acid or ester value).

Figure 14 illustrates a typical laboratory relationship of the chemical and electrical changes in mineral transformer oil, plotted as a function of the time of oxidation at 75 C., with free exposure to air. The potential sludge-formation value in its increase as a function of the time of continued oil oxidation or in relation to the increasing values of the more commonly considered chemical properties of the oil, appears to be of significance as a criterion of oil quality. The importance of the problem warrants further consideration of the potential sludge-formation test.

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[3] F. M. Clark, "Studies in the Oxidation of Mineral Transformer Oil," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 507 (1938).

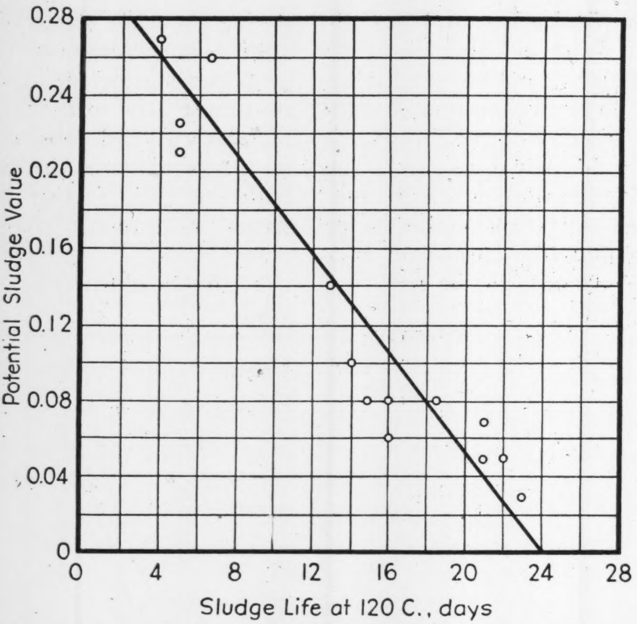

FIG. 15.—The Potential Sludge Value of Mineral Transformer Oil Bears a Linear Relation to the "Life" of the Oil as Determined by the Length of Oxidation Before the First Trace of Sludge Deposition Is Observed when the Oil Is Heated in Glass Beakers at 120 C.

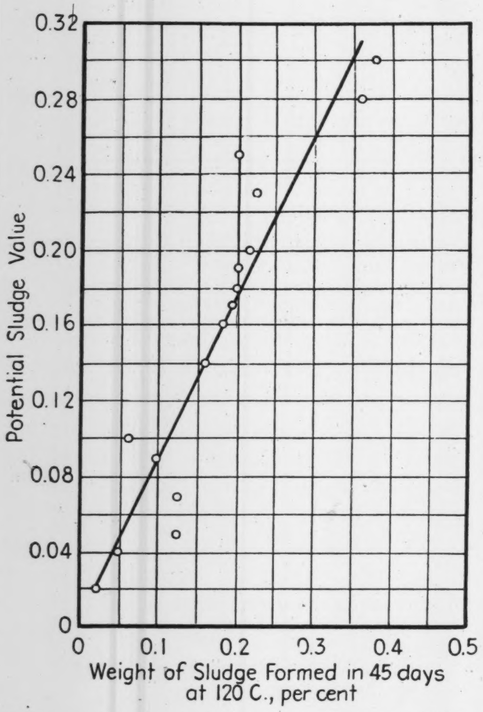

FIG. 16.—The Amount of Sludge Formed in Mineral Transformer Oil Oxidized at 120 C. in Open Beakers for 45 days Bears a Linear Relation to the Potential Sludge Value.

A variety of tests to determine the potential sludge-formation characteristic of mineral transformer oil have been suggested. In the work of the Society attention was given, first, to the time to produce the first traces of sludge formation at 120 C.[4] and more recently to the rate of sludge accumulation at 120 C. over a period of time as long as 55 days.[5] Because of the abnormal effect produced on the so-called "life" of the oil, by the presence of extremely small amounts of accidental contaminants unavoidably present in commercial practice, the first test has been abandoned in favor of the rate of sludge accumulation test.

The results obtained during oil oxidation indicate a linear relation between the oil "life" and the sludge accumulation test results and the potential sludge-formation value as determined by the high pressure oxidation procedure. The relations established are indicated in Figs. 15, 16 and 17.

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[4] E. A. Snyder, "Methods for Determining Sludging Properties of Transformer Oils," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 448 (1923).

[5] F. M. Clark and E. A. Snyder, "Testing for Sludge Formation in Mineral Transformer Oil," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 568 (1936).

The long-time sludge accumulation test is undoubtedly of value in studying the quality and condition of transformer oil in service. However, the difficulties involved in obtaining closely controlled conditions of oxidation over a period of time as long as 55 days, together with the excessive evaporation of oil and its oxidation products have proved the test to be of difficult reproduction by different operators in different laboratories. These difficulties have been largely eliminated in the high-pressure oxidation procedure used in the present analysis. No abnormal effects caused by the use of the higher oxidation temperature and oxygen pressure have been observed. The test appears worthy of further consideration.

The Power Factor Test:

The power factor of a mineral insulating oil is a characteristic which increases as the oxidation of the oil proceeds. Experience has shown, however, that the power factor of the oil is greatly affected by small quantities of conducting or semiconducting oil-soluble contaminants. Attempts to utilize the power factor test results as a criterion of oil quality and chemical condition must always be made with the clear recognition that oil-dissolved substances may be present which seriously affect the power factor

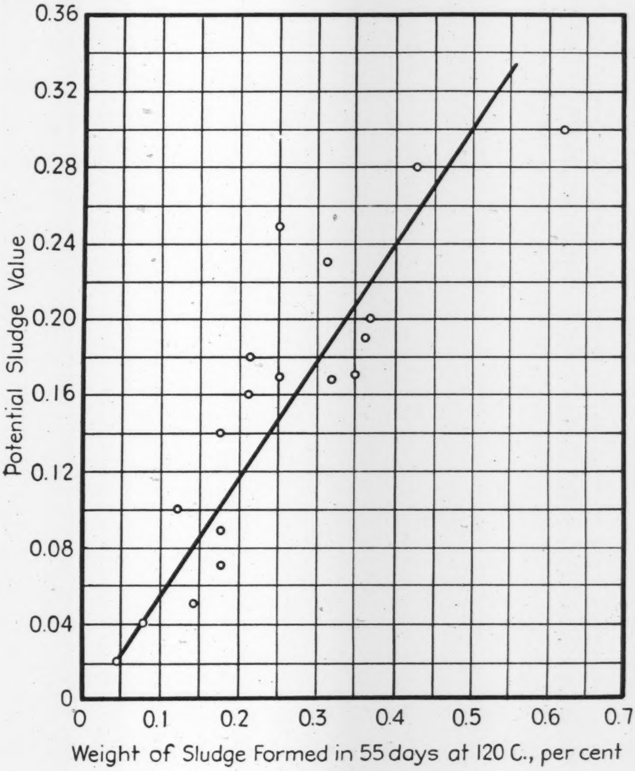

FIG. 17.—The Amount of Sludge Formed in Mineral Transformer Oil Oxidized at 120 C. in Open Beakers for 55 days Bears a Linear Relation to the Potential Sludge Value.

of the oil without corresponding effect on the dielectric strength or its chemical stability.

From a strictly laboratory standpoint where the presence of oil-soluble impurities is carefully controlled, the change in power factor values during the oxidation of mineral transformer oil is closely related to the corresponding change in the chemical properties of the oil such as, for example, that of potential sludge formation.

In Fig. 18 is illustrated the relation established between the potential sludge value of the oil and the 100 C., 60-cycle power factor. The values are measured during an oxidation run at 75 C. over a period of 300 days, the oil being maintained in glass containers allowing free air contact with the oil surface. During the oxidation of the oil, examination was made of the effect produced by the presence of zinc, copper and black varnished cloth. The results obtained clearly

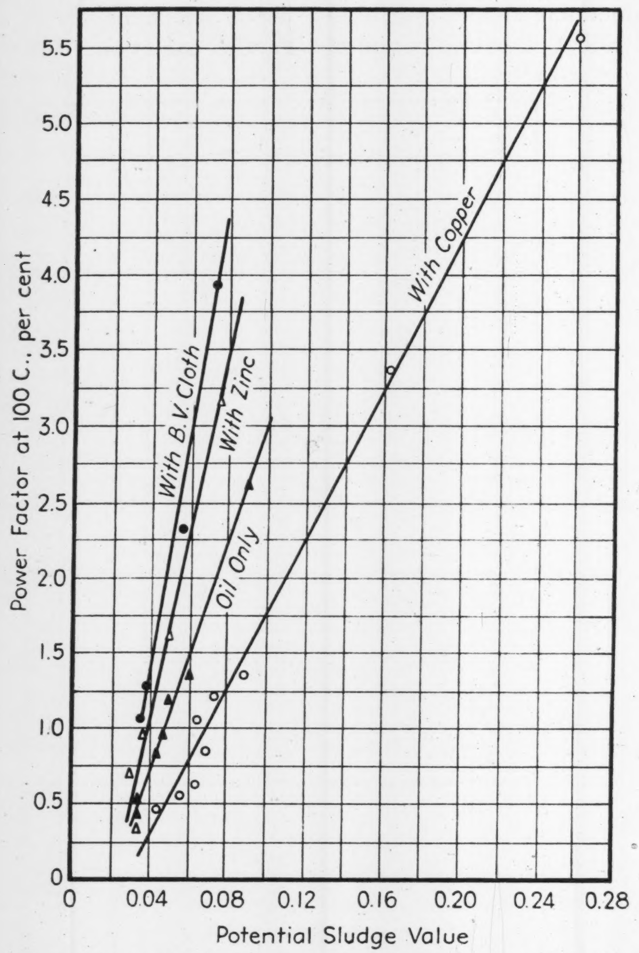

FIG. 18.—Under the Conditions of Laboratory Oxidation in Open-to-Air Glass Containers at 75 C. the Increasing Oil Power Factor Bears a Linear Relation to the Potential Sludge Value.

indicate a linear relation between the increasing tendency of the oil to produce sludge and the increase in oil power factor. The use of the power factor as a criterion of the chemical condition of the oil, however, is obscured by the fact that the power factor increase in relation to the increase in the potential sludge-formation value is greatly affected by the presence of contaminating materials. In the presence of metallic zinc, a metal chemically corroded by the oxidized oil, the power factor increases at a faster rate in relation to the potential sludge formation value than it does when the oil is oxidized alone. In the presence of metallic copper the power factor of the oil increases at a slower rate in relation to the potential sludge formation value. The result is that for a potential sludge value of 0.08 per cent the power factor of the oil varies from 4.4 per cent when the oil is oxidized in the presence of black varnished cloth, to a value 1.2 per cent when a strong oxidation catalyst such as copper is present. This behavior leads to the observation that although a linear relation exists between the power factor and the potential sludge-formation value of the oil as affected by oxida-

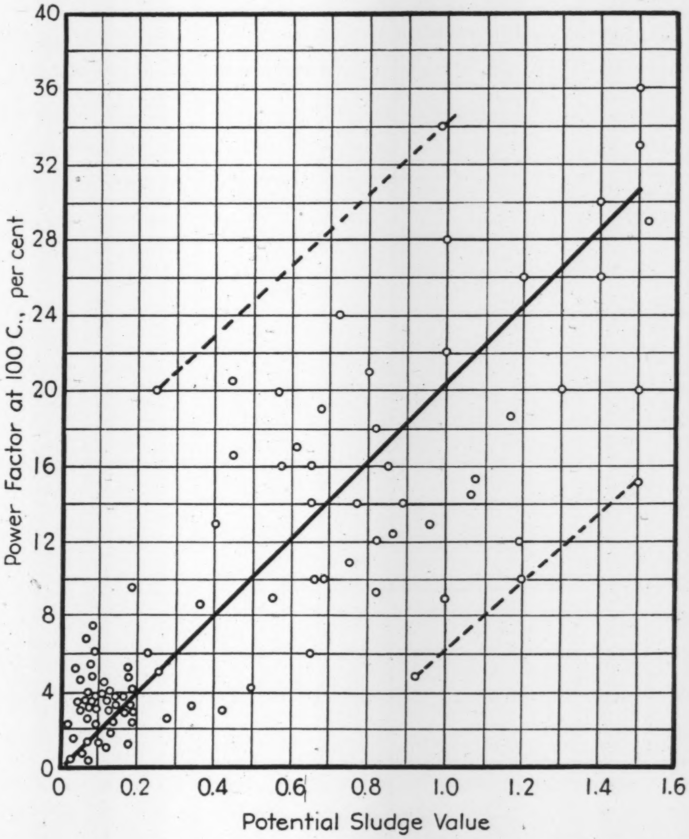

FIG. 19.—The Linear Relation Between the Power Factor and the Potential Sludge Value of Mineral Oil Drawn from Operating Transformers Is Obscured by Uncontrolled Factors Inherent in Commercial Transformer Operation.

tion at 75 C., the increased rate of oxidation and of sludging caused by the presence of a strong oxidation catalyst does not necessarily result in a corresponding increase in the oil power factor.

In the practical application of the power factor test as a criterion of oil quality and condition the combined effect of materials such as are individually illustrated in Fig. 18, together with uncontrolled variations inherent in commercial practice, tend to obscure the linear relation shown to exist between the power factor and the potential sludge-formation value. The relation as determined from tests made on mineral oil samples drawn from commercially operating transformers is illustrated in Fig. 19. The wide range in test values prevents the general acceptance of oil power factor as a gage of the quality or condition of transformer oil in commercial use except in so far as the dielectric loss is in itself of technical importance.

The Dielectric Strength Test:

The one property of mineral transformer oil which is considered of fundamental importance in successful transformer operation is that of the dielectric strength. In so far as water is a product of oil oxidation, it may be said that the dielectric strength of the oil is a function of oil oxidation since the presence of water dissolved or suspended in oil does lower the dielectric strength of the oil, especially in the presence of suspended dust or cellulosic fibers.[6] Since, however, transformers so designed as to allow the free oxidation of the oil, are also constructed in such a manner as to allow the evaporation of the water formed, there appears to be no relation between the dielectric strength of the oil and those oil characteristics generally associated with oil oxidation. This is illustrated in the data of Fig. 20 in which an attempt is made to correlate the power factor and dielectric strength values ob-

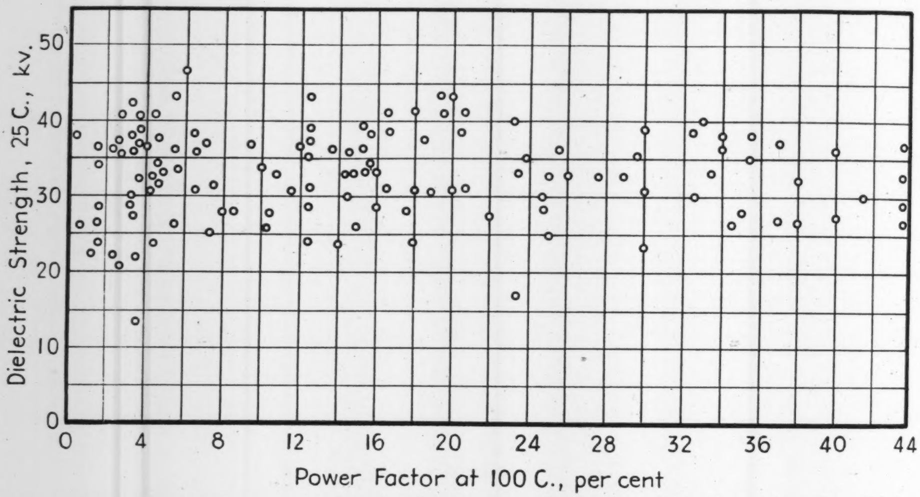

FIG. 20.—The Power Factor of Mineral Oil Drawn from Commercially Operating Transformers Bears Little Relation to Its Dielectric Strength.

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[6] F. M. Clark, "Water Solution in High Voltage Dielectric Liquids," *Technical Paper 40-77*, Am. Inst. Electrical Engrs. (1940).

tained on mineral transformer oil drawn from transformers during commercial use. Despite the dielectric change in the oil as represented by the increased power factor, the dielectric strength is not materially affected.

Attempts to correlate the dielectric strength of oil with the increase in oil acidity, potential sludge value, oil color and oil ester value have likewise been unsuccessful. The nonrelating data are eliminated for reasons of brevity. The dielectric strength of the oil is a property substantially unaffected by direct oxidation effects although the increased solubility of water and the greater tendency toward emulsification in the oxidized oil promotes the possibility of dielectric breakdown in the presence of water and secondary impurities.

The Nature of the Oil Tested—Highly Refined Oil:

The problem in gaging the condition and quality of mineral oil by any single test is made more difficult by the fact that insulating oils are obtained from a variety of crude oil sources. Each responds to oxidation in a manner dependent on its own chemical composition. Another important chemical complication is introduced by the application of different degrees of refining treatment in the preparation of oil even from the same crude oil source. The mineral transformer oils in general use may be divided into two broad classifications, the "normally" refined oil which has an iodine value in the range from 10 to 20 and the highly refined oil, sometimes called "water-white" oil which has an iodine value of substantially zero. In the latter class may be found the British A class of mineral transformer oils. In the former classification are the mineral transformer oils in general use in American practice. The analysis of the preceding paragraphs is based on the behavior of the "normally refined" American transformer oils, derived from Gulf Coast crude oil and possessing the following general properties:

Color	Less than No. 1, A.S.T.M.
Neutralization number	Less than 0.01 mg. KOH per g.
Saybolt Universal viscosity at 37.8 C	58 sec.
Specific gravity at 15.5 C/15.5 C	0.885
Iodine value	12

The general properties of the highly refined mineral transformer oil discussed in the following paragraphs are:

Color	Water-white
Neutralization number	Less than 0.01 mg. KOH per g.
Saybolt Universal viscosity at 37.8 C	80 sec.
Specific gravity at 15.5 C/15.5 C	0.870
Iodine value	0

The behavior of this latter type of oil cannot be determined with the same standards as used in the study of the "normally" refined American transformer oil although in many respects the same type of generalized relationships hold true. Unlike the less refined mineral transformer oils, the highly refined oils appear to possess a high degree of resistance to sludge formation. Instead, the oxidation products formed are strongly acidic and of a type which promotes a rapid deterioration in dielectric properties. These oils, however, present a difficult problem for laboratory analysis since many of the most corrosive acidic products formed are easily volatile under the conditions of open-to-air laboratory oxidation. These volatile products are also of a highly conducting nature, the formation of which must be evaluated in a proper study of this type of oil.

Figure 21 shows the change in the chemical and electrical properties of a highly refined transformer oil oxidized at 75 C. in open-to-air containers. It will be observed that severe chemical and electrical degradation has occurred

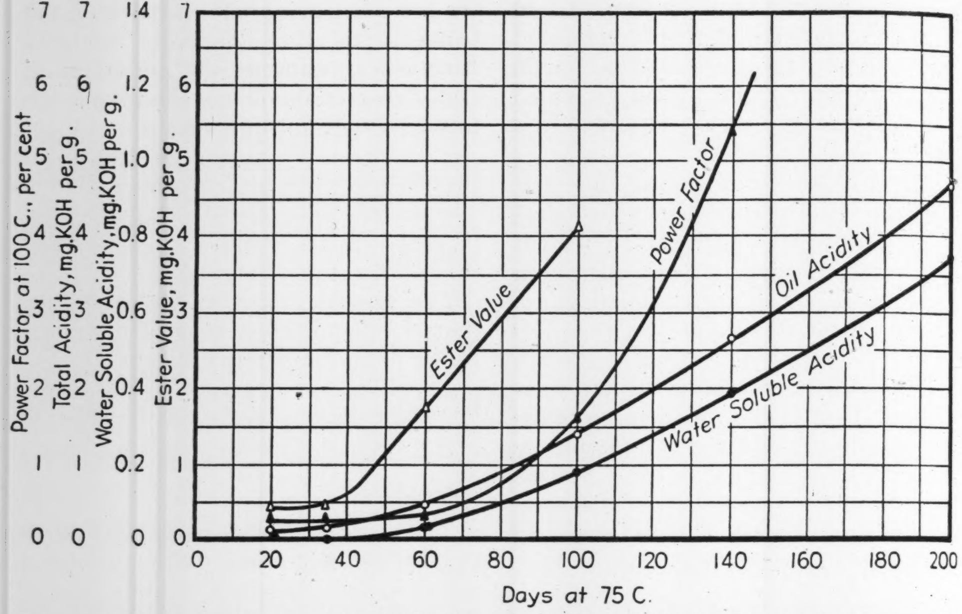

FIG. 21.—Change in the Properties of Highly Refined (Water White) Mineral Transformer Oil when Oxidized at 75 C. Under Laboratory Conditions in Open-to-Air Glass Containers.

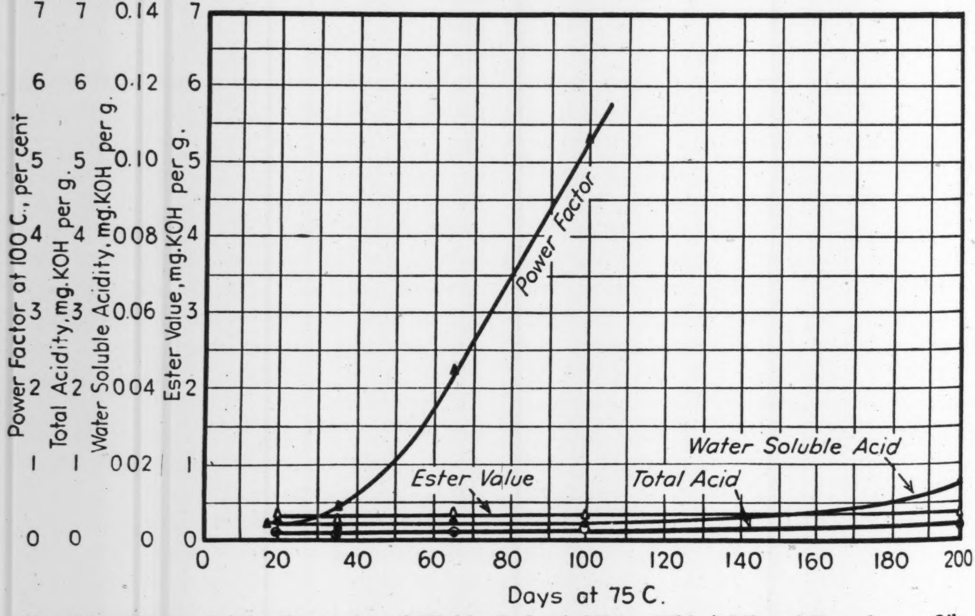

FIG. 22.—Change in the Properties of Highly Refined (Water-White) Mineral Transformer Oil When Oxidized at 75 C. Under Laboratory Conditions in Sealed Glass Containers Filled with Oil to 85 per cent of the Total Capacity.

as compared to the behavior of the "normally refined" transformer oil subjected to similar oxidation even in the presence of copper as an oxidation catalyst. Oxidized in an exactly similar manner, except that the glass containers are sealed and contain oil to 85 per cent of their volume and unconditioned air (30 per cent relative humidity at 25 C.) to 15 per cent of the total volume, the chemical changes in the highly refined oil are materially reduced. This is illustrated in the data of Fig. 22. In the latter instance, the use of a sealed container, although allowing less total oxidation of the oil because of the limited air supply, does prevent the evaporation of the oxidation products. The result is that the power factor increase in the oil oxidized in sealed containers is much more rapid despite the lesser degree of chemical deterioration as compared to the oil tests of the open-to-air oxidation run of Fig. 21. The effect is illustrated in Fig. 23. The power factor of the oil oxidized in sealed containers rises rapidly with but little increase in total oil acidity. The residual, substantially nonvolatile products retained in the oil after an open-to-air oxidation run are of less effect on the power factor of the oxidized oil. In considering the correlation of oil test properties as affected by oxidation, careful attention must be

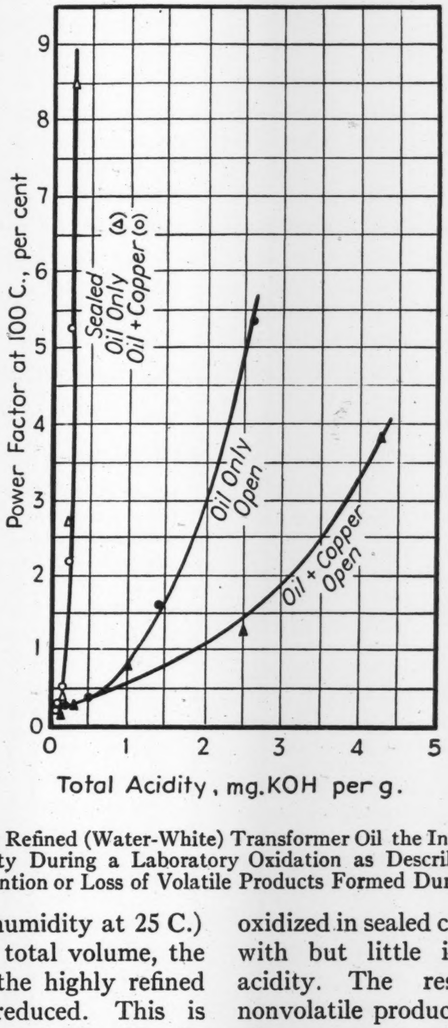

FIG. 23.—With Highly Refined (Water-White) Transformer Oil the Increase in Oil Power Factor with Increased Oil Acidity During a Laboratory Oxidation as Described in Figs. 21 and 22 Is Dependent upon the Retention or Loss of Volatile Products Formed During the Oxidation Run.

given to the type of oil under study, its degree of refining treatment, and the conditions of the oxidation run.

Is It Possible to Set Up a Single Oil Test as a Gage of Oil Quality and Condition?

At the present state of knowledge, it appears impossible to set up any single chemical or electrical test as a criterion of transformer oil quality and condition. The confusion that is often claimed to exist in this important phase of dielectric engineering is to a large part caused by the lack of proper recognition that the problem of mineral oil is neither chemical nor electrical but involves a knowledge of both properties. The electrical changes in mineral insulating oils produced as a result of oxidation reflect fundamental changes in the chemical composition of the material to a degree entirely dependent on the source of the oil, on the nature and degree of the refining treatment, and on the conditions applying during the oxidation run. For a definite type of oil, oxidized in the laboratory under carefully controlled conditions of oxidation, empirical relations can be established between the various chemical and electrical properties of the oil. From a practical standpoint, however, such empirical relations are of only general value. The severity of the transformer operation, viewed from the standpoint of possible oil oxidation, must be carefully evaluated. Color and acidity measurements constitute a good practical yardstick for general field supervision of mineral oil in commercial use, but frequent reference to standardized laboratory potential sludge formation and dielectric tests must be made in order to insure against serious and dangerous oxidation of the oil.

APPENDIX

Throughout this paper the following mineral oil testing methods have been used:

Color: Tentative Method of Test for Color of Lubricating Oil and Petroleum by Means of A.S.T.M. Union Colorimeter (D 155 - 39 T), 1939 Book of A.S.T.M. Standards, Part III, p. 598.

Acidity: Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (D 188 - 27 T), 1939 Book of A.S.T.M. Standards, Part III, p. 617.

Viscosity: Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (D 88 - 38), 1939 Book of A.S.T.M. Standards, Part III, p. 216.

Potential Sludge Value: As described in a paper by F. M. Clark on "Studies in the Oxidation of Mineral Transformer Oil," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 507 (1938). The oxidizing period was for 24 hr. at 140 C. under 250-lb. oxygen pressure in the absence of copper or other accelerator.

Ester Value: This value is obtained by subtracting the acidity value of the oil from the saponification number, both expressed in terms of milligrams of potassium hydroxide per gram of oil. The saponification value is determined in accordance with the Tentative Method of Test for Saponification Number (D 94 - 39 T), 1939 Book of A.S.T.M. Standards, Part III, p. 620.

Hydrophil: As described by K. S. Wyatt, E. W. Spring and C. H. Fellows in, "A New Method of Investigating Cable Deterioration and Its Application to Service Aged Cable," *Transactions*, Am. Inst. Electrical Engrs., Vol. 52, p. 1035-1043 (1933). The hydrophil content of the oil is expressed in area of film per gram of oil.

Resistivity: This value is determined after one minute of 500 v. d-c. application. Concentric cylinder, nickel plated brass electrodes are used, having a 0.10 in. test gap.

Power Factor: The oil power factor is measured at 100 C. under a voltage of 3000 v., 60 cycles. The test cell is exactly as described for the resistivity test.

Dielectric Strength: Standard Methods of Testing Electrical Insulating Oils (D 117 - 36), 1939 Book of A.S.T.M. Standards, Part III, p. 276.

DISCUSSION

Mr. J. H. Palmer[1] (*presented in written form*).—Mr. Clark has certainly done an excellent piece of work in correlating and analyzing the various tests now in use to determine the quality of transformer oils.

While I have had no experience with oil in connection with transformer operation, I have had considerable experience with cable oils and am discussing this paper from the point of view of a cable engineer.

Upon reading Mr. Clark's paper I found myself somewhat confused. For just what quality are transformer engineers searching in their tests of transformer oil? My impression from this paper was that the favored tests seem to be for chemical stability or instability. Mr. Clark seems to favor the test for potential sludge value, which tests the oil under conditions of accelerated oxidation only. Figure 19 shows a curve of power factor at 100 C. plotted against the potential sludge value. Mr. Clark states that "The wide range in test values prevents the general acceptance of oil power factor as a gage of the quality or condition of transformer oil in commercial use except in so far as the dielectric loss is in itself of technical importance." Judging from the curve, I should say that there is an even greater spread in the potential sludge values. Perhaps sludge formation is one of the most important criteria for the transformer oils, especially if sludge is formed in the interstices of the solid insulation. I should appreciate hearing this point explained more fully.

Our laboratories have done considerable work on stability of insulating oils for cables. Here, of course, conditions are considerably different from those encountered in transformers. In evaluating our oils, however, we have attempted to reproduce the conditions to which they are subjected in a cable, both physically and electrically, as in the various forms of bombardment tests now used. That these tests do represent cable conditions is borne out by the excellent correlation which we obtain between oil test results and results of load cycle life tests of finished cables. It seems as though some procedure of this sort could be carried out with transformer oils, subjecting the oils both to the physical conditions and electrical conditions present in transformers. It would appear then that the final criteria after aging should be electrical tests, since the transformer engineers are solely interested in whether or not the oil will withstand, for a satisfactory length of time, the electrical stress to which it is subjected.

Mr. E. J. Rutan.[2]—Mr. Clark has presented a paper which is very valuable and at the same time, to me, very satisfying. The value of Mr. Clark's paper lies in the fact that he has presented a large amount of data and through these data has shown that many of the tests which have been talked of as a measure of insulating oil properties are not of great consequence. This simplification of the testing field is exceedingly valuable and it is hoped that his paper will be referred to by many experimenters and operating engineers who wish information on insulating oils. The satisfying part of Mr. Clark's paper lies in the fact that it confirms the value of the properties which we have been measuring as having practical significance. At the time these properties were selected for operating guides we were not sure that they were the best ones to use. In Mr. Clark's paper we find confirmation for our program.

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[1] Head of Electrical Research Laboratory, Habirshaw Cable and Wire Division, Phelps Dodge Copper Products Corp., Yonkers Plant, Yonkers, N. Y.

[2] Test Engineer, Consolidated Edison Company of New York, Inc., New York, N. Y.

Mr. Clark's introduction of potential sludge value should be very helpful in further study of insulating oil; even though it is an arbitrary rating it has the possibilities, with the accumulation of data, of giving us a measuring unit for sludging value. We know it has not been possible up to the present time to agree on a suitable standard sludging test; however Mr. Clark has published his available data. It is hoped that this will serve as an encouragement to others to publish the results of their work and perhaps hasten the time when test methods can be agreed upon and interpretations of results become possible.

As one interested in the operating limits for insulating oils, I feel that it is necessary for me to criticize the presentation of the data in Figs. 8, 9, and 10. In each of these figures the oil acidity scale differs, and, this, in my opinion, has led the author to show three apparent values of acidity at which a runaway sludge condition may exist. If all of these data had been plotted on one figure to the scale used in Fig. 9 the only runaway value would have been shown to exist at an oil acidity of 3.0. The data in Figs. 8 and 10 would have served to confirm the shape of the lower portion of the curve. This criticism is advanced because those companies who are using acidity measurements as a guide in determining oil conditions might be seriously misled if they used Fig. 8 which indicates that at an acid value of 0.5 there would seem to be a danger of excessive sludge formation. At the present time in our own company we are using a value of 0.7. This value has been picked by experience and we do not have many sludge problems.

Up to the present time we have been using as operating controls the acidity, color, and dielectric strength of the oil. We are now equipping ourselves with apparatus for making sludge determinations. It would certainly be desirable, as I mentioned before, that other individuals publish their data so that the accumulated experience of several laboratories may be interpreted into the best operating practice.

MR. WILLIAM G. HORSCH.[3]—As producers of oil we are naturally very much interested in anything we can learn from the people who make or use the transformers. Mr. Clark has presented an interesting series of relationships between pairs of properties of the oil that are affected by oxidation. We have run a few transformers over a period of several years with two or three different oils in them, and while we have not covered all of the relationships that Mr. Clark has in his work, nevertheless we have checked some of them, and our findings in general are consistent with his curves.

It probably is presumptuous for oil people to say they agree with conclusions in a paper like Mr. Clark's, which contains so much field data, since they are not in a position where they are qualified to do so, but as far as I can understand the implications of his work or his data I am inclined to agree with the conclusions he has expressed. His statement at the bottom of page 1230 that the oil power factor is not a gage of quality seems particularly pertinent.

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[3] Chemist, Socony Vacuum Oil Co., Paulsboro, N. J.

It may be of interest to note in connection with Fig. 2, where Mr. Clark has plotted the power factor as a function of resistivity, that if the log of the power factor is plotted against the log of resistivity, the result is very closely a straight line, which makes a convenient relationship for quickly converting from one to the other without plotting too many points.

As regards dielectric strength, our experience checks with Mr. Clark's, namely, that in the absence of water, breakdown does not decrease with aging, even to acidity as high as 1 mg. KOH per g.

Finally, I should like to ask whether in Fig. 5 the evaporation was compensated by the addition of make-up oil.

MR. F. M. CLARK[4] (*Author's closure*). —Because of the importance of heat accumulation and heat dissipation in the successful operation of high-voltage, oil-filled cable, the cable engineer is compelled to emphasize the necessity of a low and stable power factor characteristic in the oil-treated insulation of the cable. He is assisted in this by the fact that there is normally but mild oxidation in the cable oil during service use and by the fact that the limited number of materials used in cable manufacture reduce the chance of oil contamination and erratic power factor behavior. In the transformer this is not the case. A large number of materials are used in transformer design, each of which has some effect, great or small, on the chemical and electrical stability of the mineral oil. The function of this oil is in part electrical, but to an equal degree its function is to provide a means for heat dissipation. A high loss oil does make heat dissipation more difficult, but actual experience has shown that the problem is more greatly dependent on the proper flow of the cooling oil through the oil ducts and channels provided for this purpose. This flow is prevented or reduced by sludge formation in the oil. It is for this reason that oil oxidation and sludge formation have been so exhaustively studied by the manufacturer and user of transformer oil. As shown in the paper, oil power factor appears to have some laboratory significance in its relation to oil oxidation. Eventually it may be recognized as of value even in the commercial use of the oil, although at present the vagaries of its behavior do not permit its use as a criterion of oil condition.

Mr. Palmer's suggestions are worthy of serious consideration. My own feeling is that the chemist and engineer interested in transformer oil are doing just about what the cable engineer is doing and are obtaining just about the same degree of progress.

Mr. Rutan's observations and criticism of the presentation of the data in Figs. 8, 9, and 10 are welcome. It is true that the larger the scale by which any data are plotted, the less detail one can observe of the behavior studied. The study of technical data must carefully consider this fact. In presenting the data as in Figs. 8, 9, and 10, the author has adopted a method of presentation which he believes brings out justifiable differences in the behavior of the oils, differences which are believed to be worthy of consideration in the proper evaluation of the problem under discussion.

Mr. Horsch appears to be inexcusably modest. His work in the refining of mineral transformer oils and his studies of oxidation effects have been of importance and of real assistance in the work of Subcommittee IV, of the Society's Committee D-9 on Electrical Insulating Materials. The fact that Mr. Horsch's investigations permit him to accept the conclusions of the paper under discussion is especially gratifying. In answer to Mr. Horsch's query, it is to be noted that in the "open-to-air" oxidation runs such as those of Fig. 5, oil evaporation was not compensated by the addition of make-up oil.

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[4] Chemist, General Electric Co., Pittsfield, Mass.

The more one studies the behavior of mineral oil in transformer use, the more one becomes convinced of the danger of applying any set value for any specific oil property as a criterion for safe usage. Transformer design, continuity of use, load factor and the materials of construction in contact with the oil are factors which determine not only the degree of oxidation but to a large extent the type of products formed. In our present state of knowledge, many factors must be considered before final conclusions can be drawn with regard to the usability of any particular mineral transformer oil.

THE STANDARDIZATION OF DUROMETERS

By Lewis Larrick[1]

The hardness test is one of the most useful and convenient physical tests which can be made on vulcanized rubber. It is easily made because the equipment can be simple, compact, and portable. It is particularly useful in that the test sample may be a production article which can be put into service, undamaged, after the test. Consequently the test appears in the industry in several modifications, and the tester in diverse styles.

Several more or less readily portable hardness testers are available commercially. The Shore Instrument and Manufacturing Co. has its line of "Durometers," types A, B, C, D, etc.; Schopper, through Testing Machines, Inc., has marketed a tester similar to the Shore type A; the U. S. Gauge Co. has an instrument of the same type using the case of their pressure gage; the Firestone Penetrometer is another well-known tester of the spring-load type, as is the Adams Densimeter. At the present time the Society's Committee D-11 on Rubber Products is studying four portable hardness testers which are based on the specifications of the Standard Method of Test for Hardness of Rubber (D 314 - 39),[2] three of these being spring-operated instruments, the fourth, a dead-load type. In addition to the testers mentioned above, there is the Pusey-Jones Plastometer, which is a dead-weight hardness tester rather than a plastometer.

Generally speaking, portable hardness testers are spring-operated. Beyond this feature they have little in common with each other or with either the A.S.T.M. tester (D 314) or the Pusey-Jones Plastometer (D 531 - 39 T).[3] Figure 1 gives an indication of the disparity of results obtained with some of these instruments.

The situation is little better when a comparison is made of several durometers made by the same manufacturer, as can be seen in Fig. 2, showing the hardness of one sample of rubber as measured by 20 instruments which were in use in one factory. If the hardness of this compound were specified as 71 ± 2, which is a common tolerance, the sample would have been rejected by seven of the durometers.

Further indications of the lack of agreement among durometers may be found in the discussions of the Society's Committee D-11. It is evident that a technique of standardization is necessary and that the fundamental principles should be known to designers and users. This paper presents a resumé of the theoretical laws of penetration of a material such as vulcanized rubber, and their application in standardizing Shore Type A durometers as an example of one of the widely used hardness testers.

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[1] Physical Research Laboratory, The B. F. Goodrich Co., Akron, Ohio.

[2] 1939 Book of A.S.T.M. Standards, Part III, p. 347.

[3] *Ibid.*, p. 855.

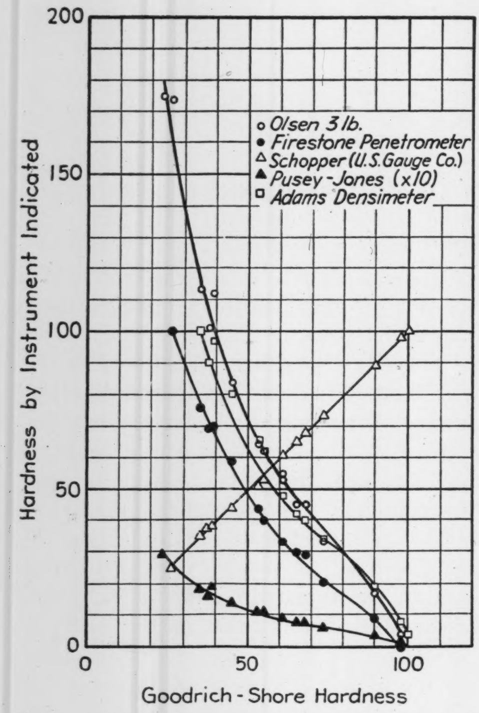

FIG. 1.—Interconversion of Hardness Readings.

Suggestions are made for an improved type of hardness tester.

THEORETICAL BASIS

It has been found[4] that the penetration of vulcanized rubber by rigid spherical indentors follows the laws of classical elasticity for indentations as high as 10 per cent of the diameter of the indenting sphere. At 20 per cent the measured penetration is not much greater than that predicted by the theory. Theory and experiment show that:

1. If a rigid spherical indentor is pressed into an isotropic elastic material, the penetration d is proportional to the two-thirds power of the load L and inversely proportional to the cube root of the diameter D of the indentor, the proportionality factor containing the elastic moduli. For vulcanized rubber, where Poisson's ratio is $\frac{1}{2}$, Young's modulus E can be determined by the relation:

$$d = (0.86/E^{2/3})\,(L^{2/3}/D^{1/3})\ldots\ldots(1)$$

2. The normal deflection of the surface of the rubber does not fall off rapidly with distance from the indentor, being 4 per cent of the maximum indentation at a distance from the indentor equal to ten times the radius of contact $a\ (= \sqrt{(dD/2)})$. Therefore the indentor should be placed several times this distance from a free edge.

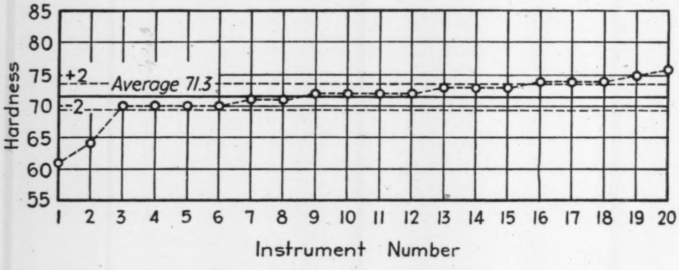

FIG. 2.—Comparison of Representative Type A Shore Durometers.

3. The compressive stress along the axis of penetration has dropped to 1 per cent of its initial value in a distance equal to ten times the radius of contact. Consequently the thickness of the sample should be this much.

4. The stress system at the center of

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[4] Lewis Larrick, "The Hardness of Rubber and Other Highly Elastic Materials," *Physical Review*, Vol. 57, p. 358 (1940).

contact is one of pure hydrostatic pressure.

5. The normal compressive stress, p, in the contact area at a distance, r, from the center is

$$p = 3L\,(a^2 - r^2)^{\frac{1}{2}}/2\,\pi\,a^3 \ldots\ldots (2)$$

where a is the radius of contact area. This is a maximum at the center and vanishes at the edge of contact.

The problem of penetration by a rigid cylinder was worked out early in the development of Elasticity Theory. If a rigid right cylinder is pressed into a plane surface, the indentation is proportional to the first power of the load and inversely proportional to the first power of the diameter, the proportionality factor containing the elastic moduli. The normal compressive stress on the face of the cylinder

$$p = L/2\,\pi\,a\,(a^2 - r^2)^{\frac{1}{2}} \ldots\ldots (3)$$

becomes infinite at the edge instead of dropping to zero as it does with the spherical indentor.

THE STANDARDIZATION OF SHORE DUROMETERS, TYPE A

Shore has stated[5] that his specifications are as follows: "This instrument, in the absence of other specific demands made upon it with respect to calibration of the dial, has been originally provided with a simple metric scale. It has a frustum cone impresser pin $\frac{3}{64}$ in. in diameter, with the flat tip $\frac{1}{32}$ in. The pin begins to yield and the indicator hand departs from zero after a pre-pressure of 2 oz., then proceeding up to 29 oz. at which point 100 is indicated on the dial. The total stroke of the pin is 0.1 in. so that each division on the dial would stand for 0.001 in. of depth travel, which while not regularly shown would read inversely, that is, the zero for the depth travel would be at 100 on the scale, and 100 depth would be at zero of the adopted scale."

The durometer is shown in Fig. 3. As the indentor *13* is pushed into the case, rotating about *8* with respect to the case, the rack *9* turns the pinion *10* and carries the pointer *5* over the scale (not shown). Consequently the scale measures directly only the motion of the pin with respect to the case This motion is resisted by the flexing of spring *1* but the reaction of the spring through linkage *4* is not in the direction of motion of the impresser pin. Since the radius of the arc of rotation of the lower end of the linkage can be varied considerably by moving slide *6*,

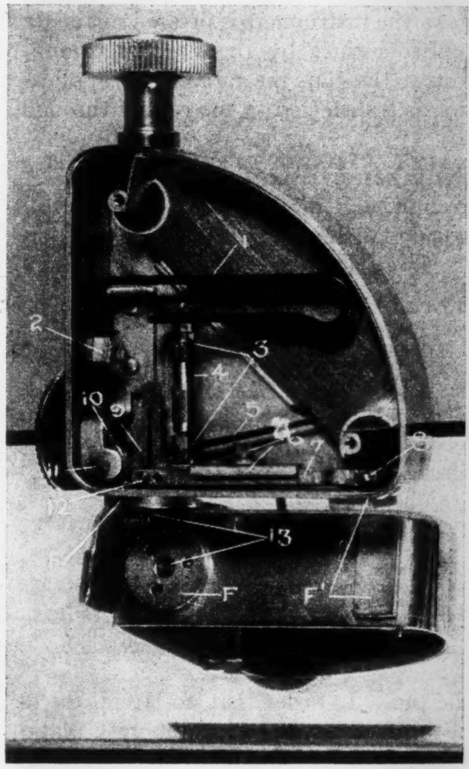

FIG. 3.—The Mechanism of a Durometer.

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[5] Symposium on Rubber, p. 96, Cleveland Regional Meeting, Am. Soc. Testing Mats. (1932). (Available as a separate publication.)

the load-deflection curve of the instrument is not necessarily a straight line. The "stress-strain" curve of the upper bearing *3* is determined by the geometry of spring *1* and by the material of the spring but the region of this curve over which deflections of the pointer take place is determined by the length of linkage *4*, once the nut *2* has been securely locked.

As the instrument is pressed against a rubber surface by the knurled thumbrest at the top, the pressure of application is transferred to the rubber through the pin. In order that no lateral forces be applied to the pin, the rest plates *F*, *F′* *must* be parallel to the surface at all times. If the pressure is greater than that just necessary to define the plane of reference the rubber will rise in the hole in *F*, carrying the indentor up with it to a falsely high hardness figure.

TABLE I.—VARIATION IN READINGS OF TYPE A DUROMETER WITH ADJUSTMENTS OF COLLAR AND SLIDE.

Adjustment	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4
1. Durometer adjusted to normal conditions	22	34	70	97
2. Slide moved 0.02 in. to left of normal	21	32	68	96
3. Slide moved 0.02 in. to right of normal	24	36	70	96
4. Slide moved 0.08 in. to right of normal	33	45	75	97
5. Slide as in 4, collar turned two faces to right	28	41	75	96
6. Collar as in 5, slide returned to normal position	19	31	69	96
7. Collar as in 6, slide moved 0.02 in. to left of normal	15	28	66	95
8. Slide as in 7, collar turned two faces to left	25	36	71	97
9. Durometer returned approximately to normal adjustments	22	34	70	96

We see there are three adjustments—position of slide *6*, length of linkage *4*, and length of pin *13*—which may be made to change the hardness reading of a durometer on a given sample. How does each of these adjustments influence the reading and the load-deflection relation? This question was answered by manipulation of a single durometer. The effect of the first two adjustments was studied by moving slide *6* to the left and to the right of normal and by rotating the collar clockwise and counterclockwise, separately and in combination. Nine settings were thus made. These adjustments gave the variations in hardnesses on four samples shown in Table I. It can be seen that large changes in hardness values of sample No. 1 were produced, but no significant change in value for sample No. 4. Because of the coarseness of the screw in the turnbuckle linkage and the roughness of the trunion arm, it is difficult to make these adjustments precisely.

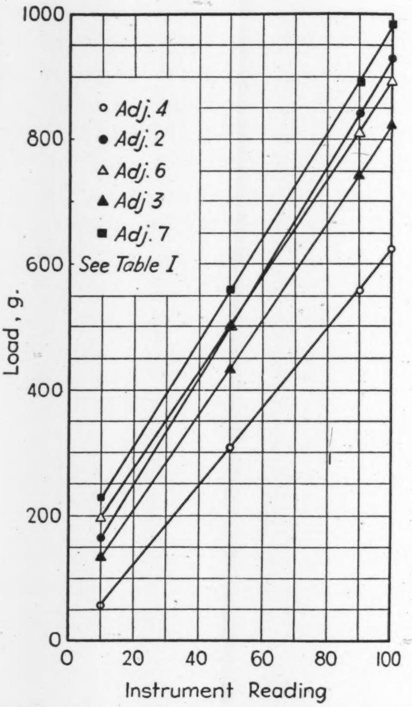

FIG. 4.—Typical Load-Scale Reading Curves.

While making these two adjustments the loads at four scale points were measured, Fig. 4. Since the curves

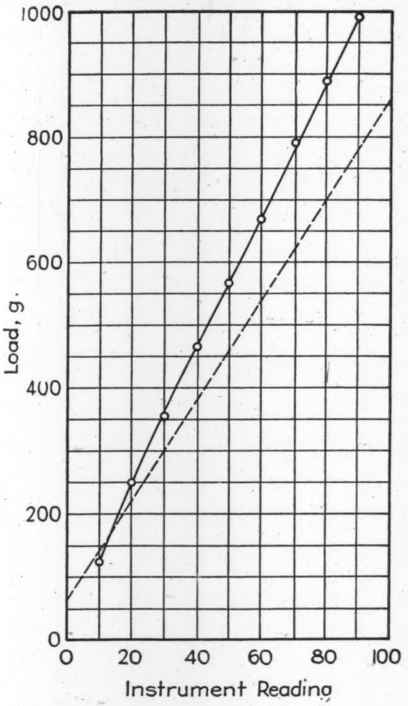

FIG. 5.—A Non-Linear Load-Scale Reading Curve.

are all of the type $y = mx + b$, it can be seen that by these two adjustments the load-deflection curve of a durometer may be moved parallel to itself (change in b) or rotated (change in m) or, generally, may be given both translation and rotation. Figure 5 shows a nonlinear load-deflection curve obtained when the slide adjustment was set far towards the left, as it has been found in durometers received for calibration.

In considering the third adjustment, it should be noted that the length of pin protruding can have no effect whatever on the load-deflection curve, though it does change the hardness reading obtained with any given sample of rubber. If d is the penetration into the rubber (length protruding), in inches, and S the corresponding scale reading it follows, from the geometry of the rack-and-pinion mechanism, that, neglecting the small departure from the straight-line motion of the pin,

$$d = K(S_a - S) = mK(L_a - L) \quad (4)$$

where:

K = a constant defined by the geometry of the mechanism,

S_a = the scale reading when the base of the pin is flush with the surface of the rest-plate,

m = the slope of the load-deflection line, and

L_a and L = the corresponding loads.

If the instrument were of the dead-load type the penetration from the

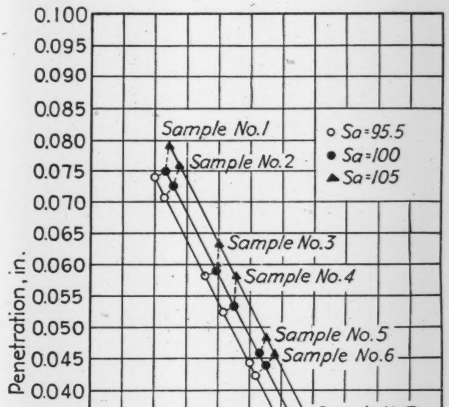

FIG. 6.—The Effect of Length of Indentor on Hardness Readings and Penetrations.

reference plane (infinite hardness) would be independent of the length of indentor. In this durometer, however, the applied load is nearly proportional to the movement of the indentor from its position at zero hardness, and the length of scale between zero and infinite hardness depends on the protruding length of indentor.

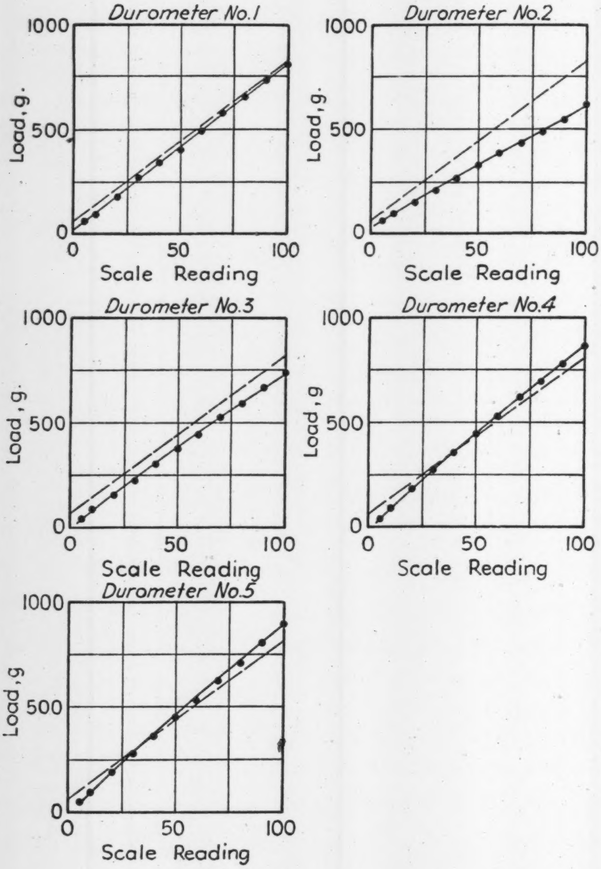

FIG. 7.—Load-Scale Reading Curves of Typical Durometers.

In order to determine the magnitude of the effect, a particular instrument was adjusted by changing the protruding length of indentor to read, successively, 95.5, 100, 105 when pressed against a flat piece of plate glass. The hardness values of a series of rubber blocks were observed for each value of S_a and the penetration calculated by Eq. 4, with the results shown in Fig. 6. These data show that the hardness reading on any sample depends on the length of pin, and the variation in hardness readings from this cause is greatest at the upper end of the scale. Therefore, this length must be specified with close tolerances.

The specifications quoted at the beginning of this section are not clear and there is no mention of tolerances. It must be inferred that a linear load-deflection curve is assumed by the manufacturer in order that the two loads given shall uniquely determine the relation. That few durometers in use comply with these simple specifications on load is shown by the data in Fig. 7. Not one of these five instruments complied with the load specifications, all five having lower than specified "pre-pressures" and three of the five having lower than normal 100-division loads.

Furthermore, the durometers met with in use do not conform to specifications in the other two respects in which specifications are given, as can be seen from Table II. It is true that the variations in length of pin given in the table are not large, *per se*, but, as the data in Fig. 6 show, a slight change in length may make a large change in hardness value, especially at the upper end of the scale. The diameters of base of indentors have been found to vary over a wide range, from 3 per cent below, to 11 per cent above, the specified value. Since the penetration is theoretically inversely proportional to diameter, there would be a wide variation in hardness readings among these five instruments, even when all are set to the same loads.

TABLE II.—OBSERVED LENGTHS OF PIN AND BASE DIAMETERS OF INDENTORS OF FIVE TYPE A DUROMETERS.

Instrument	Length, in.	Deviation from Specification, per cent	Diameter, in.	Deviation from Specification, per cent
Specification	0.09375	.....	0.03125	....
No. 1	0.0951	+1.4	0.0347	+11
No. 2	0.0933	−1.5	0.0341	+9
No. 3	0.0939	+0.2	0.0326	+4
No. 4	0.0939	+0.2	[a]	....
No. 5	0.1010	+7.7	0.0304	−3

[a] Badly rounded at edge.

As a result of the foregoing study of durometer action the following procedure for standardization was adopted by the B. F. Goodrich Co. in November, 1936:

1. The instrument is put into good mechanical condition, that is, cleaned, bearings set properly to minimize friction, pointer adjusted, parallax eliminated, and back-lash in the rack and pinion reduced as far as feasible.
2. The base of the pin is examined under a micrometer microscope. If not flat and circular the pin is either replaced or ground.
3. The load-deflection curve is adjusted to a straight line; the pre-pressure is taken as 57 g. (2 oz.) and the 100 division load as 822 g. (29 oz.) on a 0.0312 ± 0.003-in. diameter indentor. If the diameter of the base is larger, or smaller, than 0.0312 in. the loads are changed in direct proportion. A tolerance of not more than 5 g. is permitted on the load at any point.
4. The length of pin protruding from the rest plate is adjusted until, when the durometer is pressed against a flat plate glass surface, the pointer covers the 100-division mark on the scale.
5. The final check on adjustments 3 and 4 is made with the back of the case closed tightly by means of both screws.
6. During readings, the durometer is held with the rest plates F and F' parallel to the surface of the sample and pressed slowly against the sample with just sufficient force to insure contact. The initial deflection is taken as the reading.

It will be noticed that the departures from manufacturer's standards are few, but that compliance with tolerances is rigorous.

These standards have been in use at the B. F. Goodrich Co. for over three years, with approximately 70 durometers kept under control. Each durometer is returned regularly for routine inspection at least once a year. If, for any reason, the reliability of a durometer becomes questionable, it is immediately sent in for adjustment. Thus, over a period of several years, data have been acquired on the performance of the program.

TABLE III.—ANALYSIS OF TWENTY REPRESENTATIVE TYPE A DUROMETERS.

	Diameter of Face, in.	Hardness								Condition of Face of Indentor
		Sample No. 1		Sample No. 2		Sample No. 3		Sample No. 4		
		Before	After	Before	After	Before	After	Before	After	
No. 1........	0.0320	22	22	37	35	74	70	96	96	Flat, small nicks, edge rounded slightly.
No. 2........	0.0311	25	23	37	35	72	70	98	96	Flat, edge rounded slightly.
No. 3........	0.0271	29	23	39	35	71	70	96	97	Flat, edge rounded slightly.
No. 4........	0.0304	28	23	39	35	73	70	100	96	Flat, edge sharp.
No. 5........	0.0289	25	22	35	35	64	70	80	96	Flat, edge rounded slightly,
No. 6........	0.0303	23	22	36	35	70	70	97	96	Flat, edge sharp.
No. 7........	0.0314	23	22	34	35	72	70	100	96	Flat, edge sharp.
No. 8........	0.0313	24	22	34	35	70	70	97	96	Flat, edge rounded slightly.
No. 9........	0.0305	26	21	37	34	71	70	97	96	Flat, edge sharp.
No. 10.......	0.0322	24	22	37	34	73	70	101	97	Flat, edge sharp.
No. 11.......	0.0296	28	22	39	35	73	70	98	96	Out of round, edge rounded.
No. 12.......	0.0306	27	21	42	34	76	70	99	96	Edge turned over in places.
No. 13.......	0.0323	28	22	38	35	72	70	98	96	Edge rounded, surface pitted.
No. 14.......	0.0303	25	22	36	35	70	70	99	96	Edge rounded considerably.
No. 15.......	......	31	22	42	35	74	70	95	96	Diameter greater than specification, edge rounded.
No. 16.......	0.0341	24	23	37	35	74	70	99	96	Diameter greater than specification, edge rounded.
No. 17.......	0.0349	23	22	33	35	70	70	98	96	Diameter greater than specification, edge rounded.
No. 18.......	0.0334	25	22	36	35	72	70	98	96	Diameter greater than specification, surface rough.
No. 19.......	0.0280	25	22	39	35	75	70	98	96	Surface rough and pitted, edge broken.
No. 20.......	0.0292	18	22	29	34	61	70	84	96	Surface rough, out of round.

TABLE IV.—PERSONAL VARIATIONS IN STANDARDIZED DUROMETERS.

Sample	Hardness Read by Observer					
	L	Ga	K	F	S	G
A..........	36	37	36	37 to 38	37 to 38	36
B..........	33	34 to 35	35	34 to 35	35 to 36	33
C..........	38	38	39	40	40	37
D..........	41	41	41	41	41	40
E..........	55	55	54	53	55	53
F..........	38 to 39	41	41	....	....	40
G..........	58	58 to 60	57	....	....	57
H..........	45	45	45	....	45	45

The analysis of the first 20 durometers which were adjusted is illustrative of the value of this work. It will be noticed (Table III) that the data are divided into two groups. The ten durometers listed above the dividing rule in the table were *standardized* according to the procedure given above, those below the rule in the table were *calibrated* by comparison with standardized durometers because of the reasons given in the last column. These durometers could have been standardized if facilities were available for grinding the indentors but this was found unnecessary.

The hardnesses given in Table III were obtained by the author. Since the factor of personal prejudice cannot be completely eliminated by any one person, these data do not constitute reliable proof that the standardizing technique is successful. Proof which is independent of the personal factor is presented in Table IV. These data were compiled by Ivan Gazdik, of this laboratory, by collecting readings from six technical men for several compounds tested with their own durometers—which were standardized by the above method. Only two of the six have had any instruction in the author's technique of reading the durometer and only one knew that any comparison was being made.

GENERAL PRINCIPLES OF HARDNESS TESTER DESIGN

Several conclusions can be drawn from this work concerning the design of an ideal hardness tester. Obviously the manufacturer must maintain definite and close tolerances on every variable (including friction) in order that his instruments shall be reproducible among themselves. Without such close manufacturing tolerances the tester will be of little value even if it conforms, in theory, to the correct principles.

The readings given by an ideal tester measure a fundamental physical property of the sample, either the elastic modulus or a related quantity. A portable tester can do this most readily by indicating the force required to produce a specified small indent. The non-portable laboratory instrument can be equipped with several weights and a range of indentor sizes for the measurement of a wide range of hardnesses.

The ideal indentor itself is spherical (ball tipped) rather than cylindrical or "conical." The flat-faced indentor produces infinite compressive stress at the edge of contact (Eq. 3), causing abnormal wear at the edge of the indentor, and making the reading sensitive to surface friction. The spherical indentor, on the other hand, receives little wear because the compressive stress drops to zero at the edge of contact. Also, the indentation varies with the cube root of the sphere diameter, rather than with the first power of the cylinder diameter, making the hardness readings with a spherical indentor less sensitive to slight variations from the specified diameter.

The penetration produced by the tester can be any convenient value up to 10 to 15 per cent of the diameter of the spherical indentor. Since existing hardness testers employ a wide range of penetrations, up to 300 per cent of the diameter of indentor, a sample whose true hardness lies at one end of such a scale is being examined at high penetration, but a hardness at the other end of the scale is observed under very low indentation. This is one of the reasons why the relation between "durometer hardness" and "shear modulus" is subject to so much controversy in discussions about rubber mountings.

The sample whose hardness is being measured must be thick enough to eliminate the effect of the supporting surface, the minimum thickness being about 10 $\sqrt{dD/2}$. With testers now available it is possible to reverse the order of hardness of two samples as given by different durometers if the softer sample is considerably thinner than the other.

The ideal hardness tester has within itself means for establishing a reference plane of penetration. In the dead-weight instrument the zero plane can be approximated very closely by the application of a load which is negligible compared to the load under which the penetration is measured. Since the suggested portable tester measures force at a fixed penetration, the reference plane is determined by a flat annular plate on the bottom of the case, around the indentor. This plate must not in any sense be a "pressure foot." Pressure applied to the rubber over an annular ring forces the rubber up into the hole, producing a false reference plane. In addition, rubber under compression is harder than normal. Consequently, the portable durometer is pressed against the sample with just sufficient force to insure contact between sample surface and reference plane.

It should be realized that the ideal hardness tester considered here will give reproducible readings on a perfectly elastic material. However, if the sample shows any elastic after-effect, such as is present in rubber, the actual hardness will depend on the rate of application of load, just as it depends on the temperature, and degree of stretch, of the sample. All of these factors must be fixed to attain agreement on even empirical "hardness" measurements throughout the industry.

DISCUSSION

Mr. E. G. Kimmich.[1]—Both Mr. Larrick and his company should be given a vote of thanks for having made this very excellent paper available. It will be of much value to the industry.

I wish to make some remarks that might be considered supplementary to what Mr. Larrick has said, regarding the relationship of hardness measurements in industrial applications. Mr. Larrick has given us definite proof that indentation instruments can be standardized, calibrated, and used in such a manner as to remove many if not all of the present objections to such measurements of rubber.

The need for such improvement is urgent as is indicated by the fact that stiffness measurements and durometer readings cannot now be depended upon to agree better than a deviation of plus or minus 20 per cent from the mean. In other words, the maximum curve is approximately 50 per cent higher than the minimum curve.

One limitation that may persist no matter how well the indentation instrument and its use are improved and standardized, is the result of the present unavoidable variation in actual stiffness throughout the thickness of relatively thick pieces due to vulcanization variables.

For this and other reasons, Committee D-11 has adopted a load deflection type of test applicable to the compression either of pieces cut from a rubber object, or on the whole piece where a non-destructive test is required. This will supplement the hardness measurements and will be very useful in the event that such hardness determinations can never be taken out of the classification of rough measurements.

Mr. Arthur W. Carpenter.[2]—It seems desirable to emphasize the point brought out by Mr. Larrick that with an indentor having a spherical face the depth of indentation is inversely proportional to the cube root of the diameter of the sphere, but with a flat circular face it varies with the first power of the diameter of the circle. Therefore, an indentation instrument having a flat indenting element is much more sensitive than the spherical type to small differences in diameter of the indentor. This is important when we realize how difficult it is to make very small indentors with a high degree of precision suitable for dealing with indentations of the small magnitude involved in the usual hardness measurements. Obviously, the flat indentor is a poor choice, since the sphere or hemisphere would not have to be maintained to nearly as close tolerances in order to attain the same degree of accuracy in the hardness measurement. In the instrument approved by the Society for testing hardness of rubber as described in Standard Method of Test for Hardness of Rubber (D 314 – 39),[3] Committee D-11 on Rubber Products chose wisely in the light of this information when use of a hemispherical point was specified.

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[1] Mechanical Goods Development, The Goodyear Tire and Rubber Co., Inc., Akron, Ohio.

[2] Manager of Testing Laboratories, The B. F. Goodrich Co., Akron, Ohio.

[3] 1939 Book of A.S.T.M. Standards, Part III, p. 347.

MR. L. V. COOPER.[4]—One thing that the author did not stress is the ideal instrument—an instrument that measures the force necessary to produce a definite indentation.

Subcommittee XVII on Rubber Products for Absorbing Vibration of the Society's Committee D-11 realizes the need for such an instrument but as yet it has none to offer. If anyone has any ideas on such an instrument it will help greatly if such ideas are brought forward or such an instrument is submitted for inspection.

I definitely want to agree with the author that durometers as they are vended are certainly not commercial instruments. At our plant we bought four instruments and after photographing the points promptly discarded the whole group because no two were alike. We did not know which was the correct instrument.

With all the work that is necessary to bring these instruments to mechanical perfection, I wonder why the Goodrich Co. continues to use a flat pointed indentor when a hemisphere is so easily reproduced and can so easily be detected when it is out of round.

After hearing Mr. Larrick's paper, I understand why Goodrich technicians are not opposed to the durometer as is the rest of the industry. They have instruments to use which are mechanically correct whereas the rest of the industry must use instruments of unknown mechanical conditions.

MR. LEWIS LARRICK[5] (*Author's closure*).—As Mr. Cooper says, the paper contains a discussion of the ideal portable hardness tester. The chief purpose of the paper was, from the author's viewpoint, to set forth the principles of design of such an instrument.

I am sure that this durometer can be designed and built if there is demand for it. The industry must, however, desire it with sufficient intensity to justify the necessary expenditure of time and money by a good instrument maker.

In the meantime, the industry has the Shore Type A durometer. Whether we like it or not, hardness is discussed in terms of this durometer. It is because of the universal use of the Shore durometer that we, at Goodrich, have made no changes in the instrument.

Several years' experience has proved that these durometers can be standardized. Regular inspection and calibration are as necessary as with any other scientific instrument. With reasonable care a durometer will hold its calibration; otherwise it is of little value.

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[4] Test Engineer, The Firestone Tire and Rubber Co., Akron, Ohio.

[5] Physical Research Laboratory, The B. F. Goodrich Co., Akron, Ohio.

THE CREEP PHENOMENON IN ROPES AND CORDS[1]

By Carlton G. Lutts[2] and David Himmelfarb[2]

Synopsis

Ropes and cords under tension undergo continual elongation. This corresponds to the creep phenomenon in metals. Where the tension is maintained for a long enough period, the rope will actually break.

Ropes are used under constant tension in a variety of ways in which care of property and safety of life are involved. Failure of rope due to creep thus assumes a certain degree of importance, and this paper describes the relation between the rope tension and the period of time required for a rope to fail due to its continual elongation.

Ropes were loaded under tensile stresses ranging from 41 to 89 per cent of the rope breaking strengths. The periods of time required for respective ropes to break due to these static tensions varied from several minutes at the maximum tension to several hundred hours at the minimum. When first loaded, the ropes stretched at a fast rate. However, as the period of tension progressed, the rates became slower; approximately 90 per cent of the total stretch occurring in the same period of time as the last 10 per cent.

Under tension, creep continues in a rope until a limiting elongation, represented by the normal elongation at the breaking point, is reached. This limiting range appears to be constant regardless of the tension in the rope.

When tension is applied to a rope or cord there results a continual stretch, and if the tension is applied by means of a loading screw, frequent adjustment of the screw must be made in order to keep the tension constant.

Upon occasions involving stretch measurements at given loads, the authors have observed that a time element also enters into the ability of the rope to withstand tension. For example, a rope loaded with a tension in the vicinity of 80 per cent of its breaking strength will stretch progressively for some time, then suddenly break.

Apparently then, tension in rope presents a problem similar to creep in metals, and it is not difficult to visualize situations where due to this creep effect, ropes and cords will break with resulting damage even though they may have been loaded considerably below their normal breaking strength.

With this in mind, an investigation was undertaken to determine:

1. The relation between applied loads and the respective time intervals for breaking a rope.
2. The rates of stretch in ropes under constant tension.

Tests on Small Cotton Ropes

Tests were first made on small twisted cotton ropes approximately $\frac{1}{8}$ in. in diameter. The ends of each of these rope specimens, which were approximately 4 ft. long, were wound on two small drums similar to the holders on a cordage strength testing machine, and one of the drums was suspended from the ceiling. The lower drum was loaded with weights to create the desired tension in the rope. After free suspension of the load, the time interval for the rope to break was observed using an electrical interval timer arrangement. Several de-

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[1] Opinions or assertions contained in this paper are not to be construed as official or reflecting the views of the Navy Dept., or the Naval Service at large.

[2] Materials Engineer, and Assistant Textile Technologist, respectively, U. S. Navy Yard, Boston, Mass.

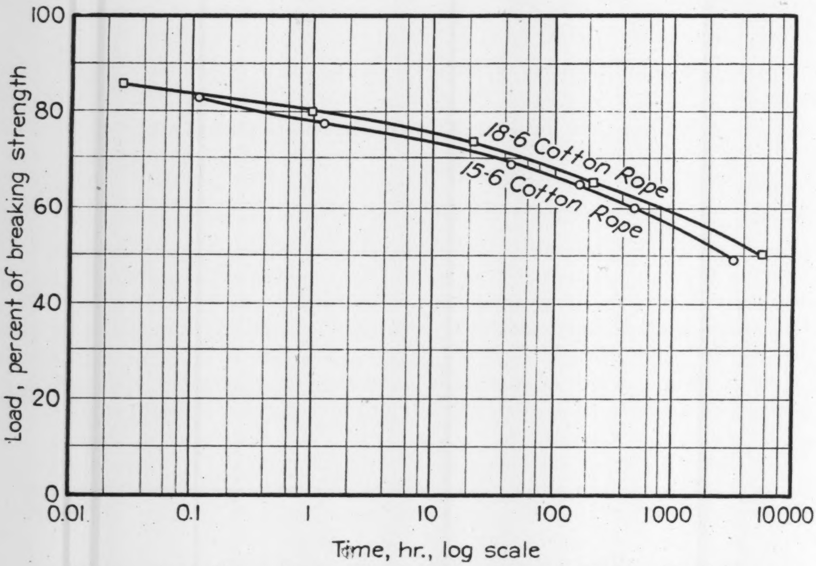

FIG. 1.—Periods Required to Break Cotton Ropes at Various Tensions.

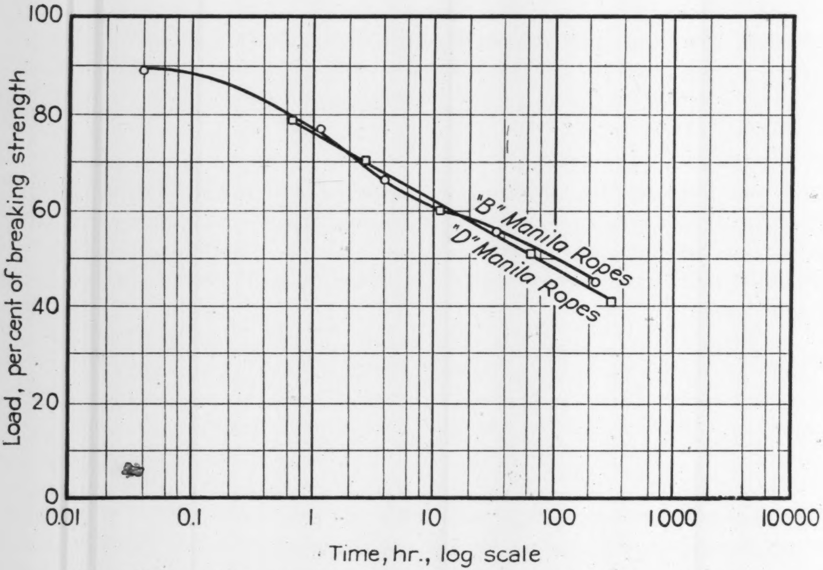

FIG. 2.—Periods Required to Break Manila Ropes at Various Tensions.

TABLE I.—PERIODS FOR BREAKING TWISTED COTTON ROPES UNDER STEADY LOADS.

Ropes Designated 15-6, Average Breaking Strength 189 lb. when Broken in Strength Testing Machine			Ropes Designated 18-6, Average Breaking Strength 201 lb. when Broken in Strength Testing Machine		
Load, per cent of breaking strength	Time Required to Break, hr.	Average Time, hr.	Load, per cent of breaking strength	Time Required to Break, hr.	Average Time, hr.
84	0.02		87	0.03	
	0.10			0.03	0.03
	0.21	0.12		0.03	
	0.13				
	0.25		80	1.0	
	0.03			2.0	
				0.7	1.0
78	2.0			0.5	
	1.5	1.4		0.7	
	0.6			1.1	
69	12		73	1	
	80			16	21
	3	47		5	
	40			63	
	132				
	12		66	48	
				488	208
65	175	175		169	
				128	
60	670				
	573	481	50	5860	5860
	243				
	436				
49	3500	3500			

TABLE II.—PERIODS FOR BREAKING ½-IN. DIAMETER MANILA ROPES UNDER STEADY LOADS.

Ropes Designated B, 28 deg. Angle of Twist, Average Breaking Strength 2820 lb., when Broken in Strength Testing Machine			Ropes Designated D, 22 deg. Angle of Twist, Average Breaking Strength 3150 lb., when Broken in Strength Testing Machine		
Load, per cent of breaking strength	Time Required to Break, hr.	Average Time, hr.	Load, per cent of breaking strength	Time Required to Break, hr.	Average Time, hr.
89	0.06		79	0.05	
	0.02	0.04		0.53	0.68
	0.03			1.47	
78	1.8		70	2.5	
	0.9			3.5	3.0
	2.2	1.2			
	0.7		60	3.6	
	0.1			20.7	12.2
	1.7				
			51	51	
67	2.6			75	63
	5.4	4.0			
			41	302	
57	4.3			345	325
	39.5	36.9		327	
	66.8				
46	364				
	207	238			
	144				

terminations were made with each load in an effort to obtain a consistent average.

The results of these tests are shown in Table I and in Fig. 1. Extrapolation of the curves in Fig. 1 shows that even while sustaining a nominal load, breaking of the ropes may be expected after a period of time.

Tests on Manila Ropes

The tests described were followed by a series of similar tests on $\frac{1}{2}$-in. diameter manila ropes which were loaded with dead weights as high as 2500 lb. These ropes were tested under constant relative humidity of 65 per cent at 70 F. Two sets of ropes were used, one termed B ropes, twisted with the normal angle of twist of 28 deg., the other termed D ropes, twisted very slack with an angle of twist of 22 deg. The test specimens were prepared by splicing the ends of a length of rope into eyes. One eye was passed through a timber of circular cross-section securely mounted near the ceiling of the room; the other eye was mounted over a sheave to which the tension weight was attached. The length of each test rope between the ends of the splices was approximately 4 ft. The results of these tests, shown in Table II and in Fig. 2, corroborate the results obtained with the small cotton ropes. Since the slopes of the curves in Fig. 2 are greater than those in Fig. 1, sustained loading of the manila ropes will result in rupture faster than corresponding tension in the smaller cotton ropes. By extrapolation of the curves in Fig. 2, a $\frac{1}{2}$-in. manila rope loaded in tension corresponding to a factor of safety of 5 (20 per cent of the breaking strength) can be expected to break in about one year as a result of the creep only, and if fiber degradation and other adverse influences set in, the life of the rope even under comparatively low sustained tension becomes relatively short.

It is interesting to note that creep resulting in actual breaking or fracture is not unusual, but has been observed in other fields. For example, in tests on lead and lead alloys described by Moore and others,[3] sustained loads of approximately 50 per cent the initial tensile strength caused rupture of the metal specimens in a period of approximately one month.

TABLE III.—ELONGATIONS TO THE BREAKING POINTS UNDER VARIOUS TENSIONS—MANILA ROPES.

	Load, per cent of breaking strength	Total Elongation, per cent
Ropes B...........	100	12.2
	89	12.1
	78	12.0
	67	13.3
	57	12.2
	46	11.2
Ropes D...........	100	8.9
	79	7.6
	70	7.2
	60	8.1
	51	7.2
	41	6.9

STRETCH OF MANILA ROPES UNDER TENSION

The B and D manila ropes used in the tests already described suffered average elongations of 12.2 per cent and 8.9 per cent, respectively, when loaded to their breaking points in the testing machine.

In Table III are shown the total elongations during the periods required for the ropes to break when loaded with the various degrees of constant tension. While these results show some variation, they tend to show that a rope under tension will stretch, and as soon as the total stretch reaches a limiting value represented by the total stretch at its normal breaking point, the rope breaks.

The amounts of stretch at various periods while the ropes are under their respective tensions are shown in Figs. 3 and 4. All these curves were plotted with the amounts of stretch, expressed as percentages of the total stretch, as ordinates.

From these curves, it will be noted that if half the total stretch is considered at a time, the first half takes place at a much faster rate than the latter half. In Table IV are shown the proportions of the total stretches undergone during the first half periods of sustained tension.

TABLE IV.—ELONGATIONS DURING FIRST HALF PERIODS OF SUSTAINED LOADINGS.

	Load, per cent of breaking strength	Time, Half-Period, hr.	Elongation, per cent of total elongation during initial half period
Ropes B.......	89	0.02	68
	78	0.6	87
	67	2.0	87
	57	18.5	87
	46	119.0	92
Ropes D......	79	0.34	87
	70	1.5	90
	60	6.1	91
	51	31.5	92
	41	162.5	92

These data are taken directly from the curves in Figs. 3 and 4.

With the exception of one rope, between 87 per cent and 92 per cent, or an average of 90 per cent of the total stretch takes place during the first half periods of sustained tension to the breaking point.

SUMMARY

When ropes and cords are loaded to tensions considerably below their normal breaking points, they are subject to continual elongation or creep.

As soon as these elongations reach limiting values represented by normal elongations at the breaking point, the ropes break.

The time required for the loaded ropes to break due to creep varies from a matter of several minutes when the load approaches 90 per cent of the breaking strength to several hundred hours at a load of approximately 40 per cent of the breaking strength.

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[3] H. F. Moore, *et al.*, "The Creep and Fractures of Lead and Lead Alloys," *Bulletin No. 272*, Engineering Experiment Station, University of Illinois (1935).

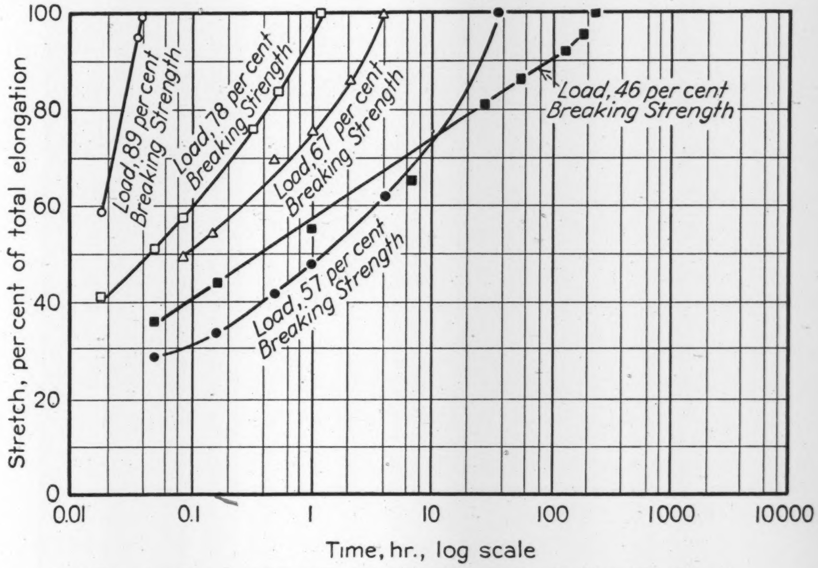

FIG. 3.—Rates of Stretch of B Manila Ropes at Various Tensions.

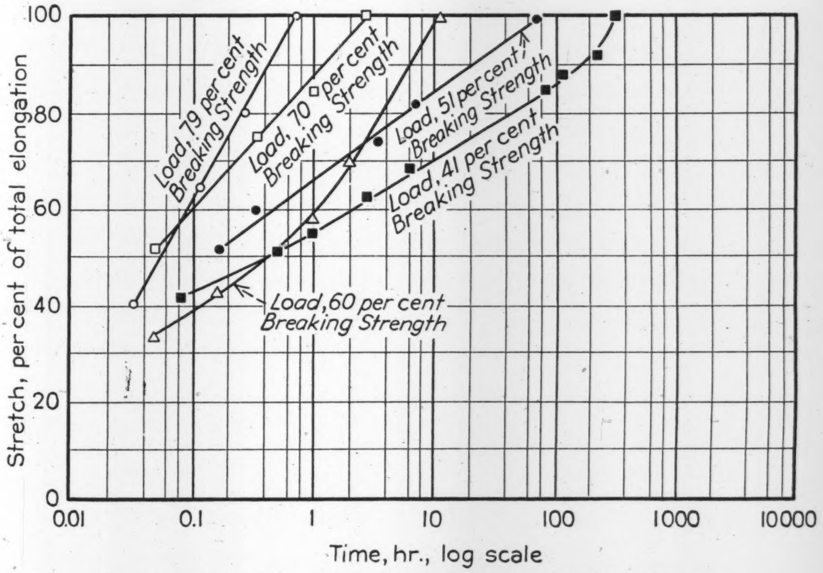

FIG. 4.—Rates of Stretch of D Manila Ropes at Various Tensions.

When first loaded, the ropes stretch at a rate greatly in excess of the rates of stretch during the latter part of the periods of sustained tension, approximately 90 per cent of the total stretch occurring in the period of time required for the last 10 per cent of the total stretch.

ACCELERATED WEATHERING OF TRANSPARENT PLASTICS

By G. M. Kline,[1] W. A. Crouse,[1] and B. M. Axilrod[1]

Synopsis

In previous reports of work on transparent plastics at the National Bureau of Standards, it was noted that accelerated aging tests made with apparatus employing an inclosed carbon arc as the source of ultraviolet light failed to correlate well with the results of outdoor exposure.[2,3] A detailed investigation of accelerated weathering of transparent plastics was, therefore, undertaken in an endeavor to obtain a reliable test for rapidly evaluating their weathering properties.

Sources of ultraviolet light which were studied included the Weather-Ometer, Fade-Ometer, Eveready Accelerated Testing Unit, Sunlamps with S-1, S-4, and H-4 bulbs, and Cooper-Hewitt quartz-tube mercury-arc lamps. Many of these lamps were operated with and without Corex D glass filters. The effect of the temperature of the samples during irradiation was also considered. The samples were exposed to the light with and without alternate cycles of wetting with water. Three different methods of wetting the specimens were employed: namely, immersion in water, spraying with water, and exposure to a mist-laden atmosphere in a closed cabinet.

The effects of these different accelerated weathering conditions upon various types of transparent plastics were evaluated by measurements of light transmission and haze, and by visual examination of the specimens for crazing, fusing, blooming, discoloration, and warping. These results were compared with data obtained for specimens of the same plastics exposed to the weather in Washington, D. C. On the basis of these tests a method of accelerated weathering of transparent plastics, involving alternate exposure to a misty atmosphere and to light from an S-1 Sunlamp bulb, has been developed which gives results correlating well with the actual behavior of the materials in outdoor exposure tests.

In previous reports of work on transparent plastics at the National Bureau of Standards, it was noted that accelerated aging tests made with apparatus employing an inclosed carbon arc as the source of ultraviolet light failed to correlate well with the results of outdoor exposure.[2,3] A comprehensive investigation of accelerated weathering of transparent plastics was, therefore, undertaken in an endeavor to obtain a reliable test for evaluating their aging properties. This work was carried on as part of a project established at the

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[1] Chief, Organic Plastics Section, Junior Physicist, and Assistant Physicist, respectively, National Bureau of Standards, Washington, D. C.

[2] B. M. Axilrod and G. M. Kline, "Study of Transparent Plastics for Use in Aircraft," *Journal of Research*, Nat. Bureau Standards, Vol. 19, pp. 367–400 (1937).

[3] G. M. Kline, "Permanence of Plastics," Symposium on Plastics, Rochester Regional Meeting, Am. Soc. Testing Mats., p. 35 (1938). (Symposium available as a separate publication.)

National Bureau of Standards by the National Advisory Committee for Aeronautics to determine the suitability of various transparent plastics for use as windshields on aircraft.

MATERIALS

The types of transparent plastics examined include cellulose nitrate, cellulose acetate, cellulose acetate-propionate, cellulose acetate-butyrate, ethylcellulose, polymethyl methacrylate, polyvinyl chloride-acetate, polyvinyl butyral, polystyrene, and glyceryl phthalate. Specimens representing materials obtained from different manufacturers as well as compositions containing various types and amounts of plasticizers were subjected to a variety of accelerated aging conditions. Other specimens of these plastics were exposed outdoors in Washington, Florida, and Panama. This report presents the results of accelerated aging tests on only a limited number of the transparent plastics studied and compares these laboratory results with those obtained by outdoor exposure in Washington.

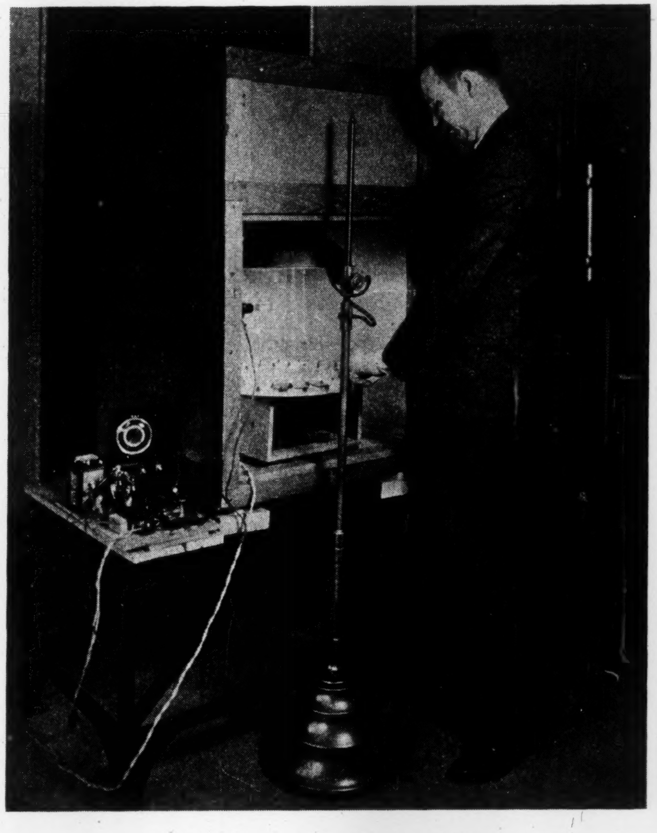

FIG. 1.—Sunlamp Irradiation Cabinet.

APPARATUS

Outdoor Exposure Tests:

Flat specimens 3 by 6 in. and of different thicknesses from approximately $\frac{1}{16}$ to $\frac{1}{4}$ in. were fastened in brass frames, with the bottom clamped tightly and the top and sides free to move in order to take care of expansion and contraction. One-fourth of the specimen was shielded from direct sunlight by a metal strip fastened about $\frac{1}{4}$ in. above the specimen. The frames were fastened to wooden exposure racks on the roof of the Industrial Building of the National Bureau of Standards, the racks facing south and being inclined at an angle of 45 deg. to the horizontal.

Accelerated Aging Equipment—Light Sources:

Sources of ultraviolet light which were investigated included the Sunlamp with S-1, S-4, and H-4 bulbs, Alpine mercury-arc lamp, Cooper-Hewitt mercury-arc lamp, Eveready accelerated testing unit, Weather-Ometer, and Fade-Ometer. Some of the lamps were operated with and without Corex D glass filters. A brief description of each of these light sources follows.

Sunlamp BM6 with S-1 Bulb (Fig. 1).—The light source is a combination tungsten-filament mercury-arc inclosed in Corex D glass which filters out most of the ultraviolet light below 2800 Å. The lamp is rated at 450 w. The test specimen is 4 in. by $1\frac{1}{2}$ in. with a tapered end. The specimens are carried on an aluminum disk which is centered under the S-1 bulb and rests on a phonograph turntable, operated at 33 rpm. Two sets of 21 machine screws and nuts attached to the disk spaced equidistantly on circles $11\frac{3}{4}$ and $15\frac{3}{4}$ in. in diameter, respectively, support the specimens approximately $\frac{3}{16}$ in. above the disk. The plane of the specimens is 12 in. from the bottom of the S-1 bulb and the portion of the specimen at which measurements of light transmission and haze are made is 5 in. from the center of the turntable.

S-4 Lamp.—The light source is a mercury arc inclosed in a quartz capillary and further enveloped in Corex D glass. The lamp is rated at 100 w. This lamp with suitable transformer was substituted for the BM6 Sunlamp in the cabinet shown in Fig. 1.

H-4 Lamp.—The light source is the same as in the S-4 lamp. Nonex glass, which filters out most of the ultraviolet light below 3000 Å, is used for the envelope. This lamp was also used in the cabinet shown in Fig. 1. In one test an H-4 lamp with the Nonex glass envelope removed was employed.

Alpine Lamp (Fig. 2).—The light source is a mercury arc inclosed in a quartz tube. This lamp is rated at 730 w. The specimens were carried on the same type of turntable used with the Sunlamp. The plane of the specimens was 10 in. from the tube. A shield of Corex D glass was inserted between the quartz tube and the specimens.

Eveready Accelerated Testing Unit X-1 (Fig. 3).—The light source is composed of two pairs of carbons; the arc shifts from one pair to the other at 20- to 30-min. intervals. Operating at 60 amp. arc current with 50 v. across the arc, the input required for this lamp on 220 v., a.c., is 3100 w. The specimens are hung in metal frames on a drum which rotates them at 2.4 rpm. at a distance of 20 in. from the arc. A shield of Corex D glass is inserted between the specimens and the carbon arc.

Fade-Ometer (Fig. 4, left).—The light source is composed of one long and two short carbons, which burn within an enclosure consisting of a globe of Corex D glass and a metal plate through which the carbons protrude. This lamp is operated on 220 v., d.c., at 13 amp. arc current with 140 v. across the arc. The specimens are hung in metal frames at a distance of 10 in. from the arc and are not rotated.

Weather-Ometer (Fig. 4, right).—The light source is the same as that of the Fade-Ometer except that only one long and one short carbons are employed.

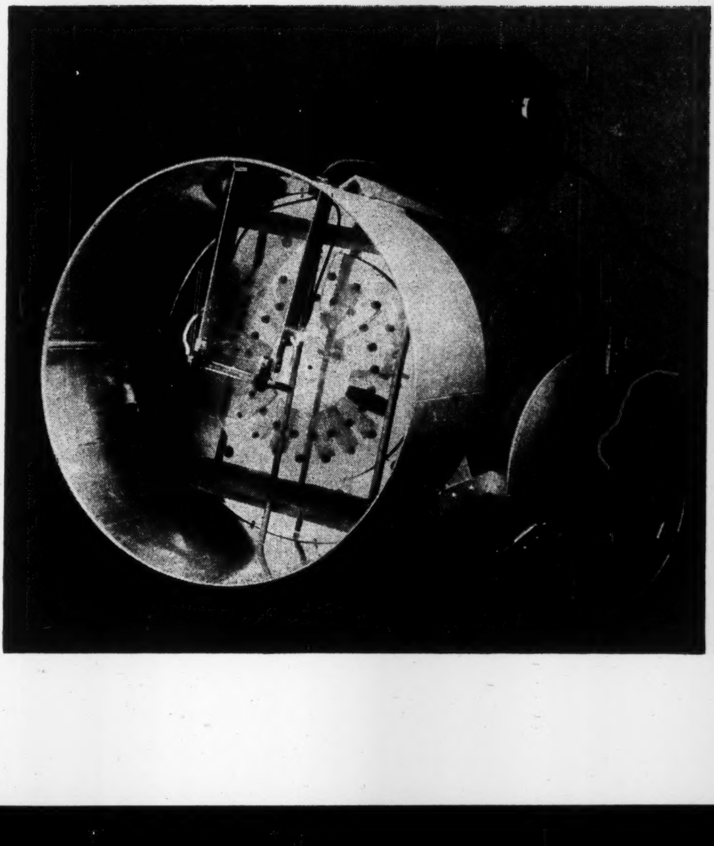

FIG. 2.—Alpine Irradiation Unit.

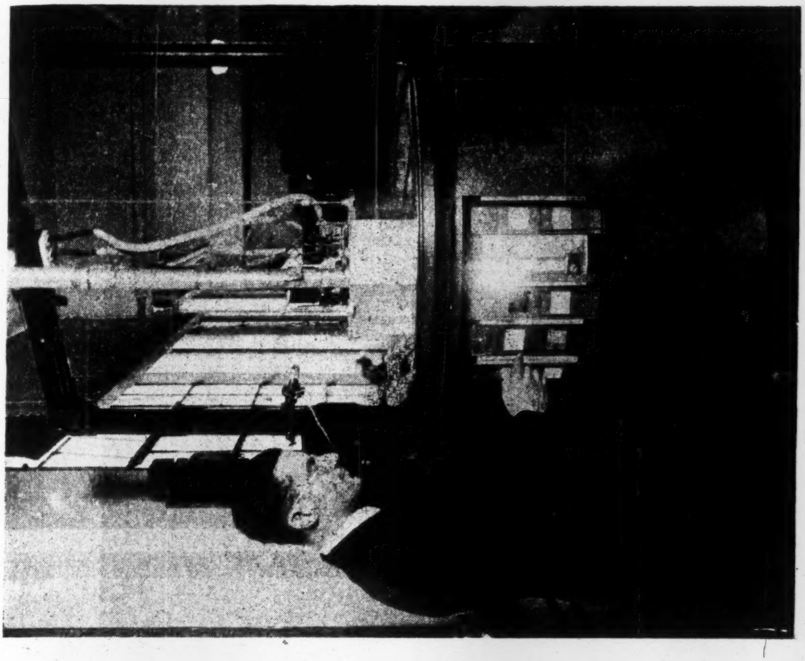

FIG. 3.—Eveready Accelerated Testing Unit.

The specimens are clamped to solid aluminum frames which are fastened on a drum 15 in. from the arc and rotated at 0.05 rpm.

FIG. 4.—Fade-Ometer (left) and Weather-Ometer (right).

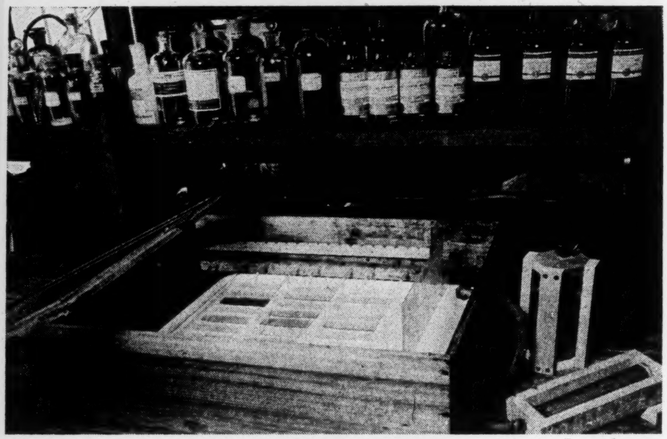

FIG. 5.—Fog Chamber.

Accelerated Aging Equipment—Fog Chamber:

The transparent plastics were exposed to the ultraviolet light with and without alternate cycles of wetting with water. Three different methods of wetting the samples were employed: namely, immersion in water, direct spraying with water, and exposure to a mist-laden atmosphere in a closed fog chamber (Fig. 5). The fog chamber consists of a shallow well-paraffined box containing an enameled pan partially filled with distilled water. The suction tube of a spray unit dips into this water and the spray is directed against a baffle which is as high as the fog chamber and about two-thirds as wide. The specimens are placed behind the baffle on glass rods which bridge the enameled pan.

Light Transmission and Haze Measurements:

The effects of the accelerated aging treatments and weather exposure upon the transparent plastics were evaluated by visual examination of the specimens for crazing, fusing, blooming, discoloration, and warping, and by measurements of light transmission and haze. The instrument used for these latter measurements was developed in the course of our work on transparent plastics and is shown in Fig. 6. This hazeometer has been previously described.[2] To obtain a measure of the light transmission of the plastic, the light is adjusted in intensity so that a current of 100 μ amp. is obtained from the photoelectric cell, the specimen is clamped against the cell at position *a*, and the decrease in current output of the cell is noted. To obtain a measure of the haziness of the plastic, the specimen is placed at aperture *b*, 18 in. distant from aperture *a*, and the current output of the cell is again read. Haziness is defined as the percentage of the total light transmitted at position *a*, which is scattered when the sample is placed at position *b*.

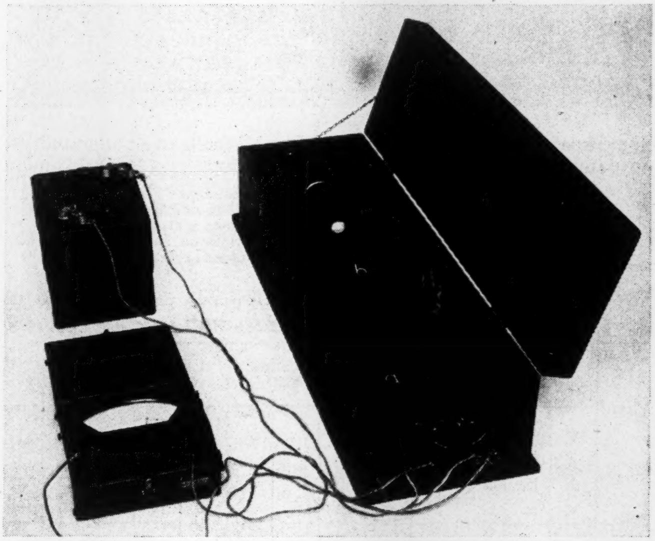

FIG. 6.—Hazeometer.

EXPERIMENTAL RESULTS

The experimental work of this investigation can be considered in three phases: (1) the preliminary work with well-known accelerated aging equipment, (2) the development of a new procedure for accelerated aging of transparent plastics, and (3) comparison of various lamps as sources of ultraviolet light for use in this new method. Details of the experiments conducted in the first phase are available elsewhere.[4]

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[4] G. M. Kline, W. A. Crouse and B. M. Axilrod, "Accelerated Weathering of Transparent Plastics," *Modern Plastics*, Vol. 17, August, 1940, pp. 49–57, 86, 88.

Development of New Aging Test:

Moisture contacts the specimens in roof exposure tests as rain and as dew, with the latter predominating in so far as number of times of occurrence is concerned. A spraying procedure would reproduce the effect of rain, but not that of dew which remains on the plastic for an appreciable length of time and then dries without any accompanying washing effects. This action would tend to bring decomposition products to the surface. Furthermore, we have observed that repeated contact of some plastics with water in the form of droplets will lead to a permanent orange-peel effect with resultant distortion of vision through the material. This effect is also observed on these plastics after a few weeks of exposure on the roof, but it is not duplicated by spraying them with water in the laboratory aging tests nor by immersion in water unless accidental collection of air bubbles causes unequal contact of the water with the plastic. On the basis of these considerations, a fog chamber was constructed in which the specimens are exposed to a misty atmosphere so that the upper surface becomes completely covered with droplets of water without being subjected to any washing action.

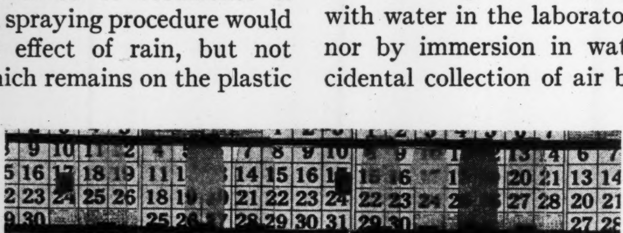

FIG. 7.—Comparison of Effects of Various Wetting Methods in Accelerated Weathering of Transparent Plastics. Source of Ultraviolet Light was the S-1 Sunlamp.

Water treatments	Specimens
A. Spray	No. 3—Cellulose acetate A7
B. Immersed	No. 17 - Cellulose acetate B68
C. Fog	No. 54—Cellulose nitrate W1
D. None	No. 56—Cellulose acetate C16

TABLE I.—COMPARISON OF VARIOUS METHODS OF WETTING SPECIMENS IN ACCELERATED AGING TESTS MADE WITH S-1 SUNLAMP.

	Cellulose Acetate Sample A7				Cellulose Acetate Sample B68				Cellulose Acetate Sample C16				Cellulose Nitrate Sample W1			
	A, Sprayed	B, Immersed	C, Fog Exposure	D, Dry	A, Sprayed	B, Immersed	C, Fog Exposure	D, Dry	A, Sprayed	B, Immersed	C, Fog Exposure	D, Dry	A, Sprayed	B, Immersed	C, Fog Exposure	D, Dry
Light transmission, per cent																
Initial	85.7	85.6	85.5	85.7	91.7	91.7	91.5	91.8	83.8	83.8	83.7	83.7	87.5	87.6	87.5	87.6
After 200 hr.	86.7	86.6	86.5	86.5	92.2	92.0	91.8	92.1	84.7	84.5	84.5	84.6	84.7	83.7	83.6	84.8
After 500 hr.	81.5	77.9	82.8	87.1	87.8	87.5	79.9	92.2	84.9	84.3	83.8	85.0	59.3	58.2	57.7	81.1
Haze, per cent																
Initial	6.1	5.8	6.1	5.5	3.5	3.4	3.5	3.7	7.8	7.8	8.1	7.8	5.1	4.9	4.8	4.7
After 200 hr.	6.0	6.1	6.6	5.1	4.0	3.7	4.5	3.6	7.8	8.3	8.4	8.2	6.0	11.2	10.9	4.8
After 500 hr.	28.0	42.4	43.0	5.7	20.6	16.2	41.4	4.0	9.4	10.9	17.7	8.5	83.5	89.7	66.4	5.6[a]

[a] Measured outside of spotted areas.

An experimental run was made to compare this new method of wetting the plastics in accelerated aging with other wetting methods. Four sets of samples were subjected to 500 hr. of ultraviolet light from an S-1 bulb under the following conditions of contact with water:

Set A. Sprayed with water four times each day for 1 hr., alternating with 1-hr. exposure to the light; run overnight without water cycles.

Set B. Same procedure as *A* except for substitution of immersion in water in place of spraying.

Set C. Same procedure as *A* except for substitution of exposure in the fog chamber in place of spraying.

Set D. No water cycles.

The results of light transmission and haze measurements on these samples are presented in Table I and the condition of the samples after 500 hr. of light exposure is shown in Fig. 7. The crazing of identical samples exposed to moisture cycles of spray, immersion, and fog, respectively, was about the same for the three types of wetting. However, only sample C-56, exposed in the fog chamber, exhibited the surface orange-peel that occurs characteristically on outdoor exposure of this particular material. The set of samples exposed only to the light was much less deteriorated than the other groups; only the cellulose nitrate specimen D-54 was crazed and then only in spots where water accidentally came in contact for a short time with the specimen.

Following the experimental run just described, six runs were made with the sunlamp - fog chamber apparatus in which a total of 120 transparent plastics of many different types and compositions were tested. A specimen of a particular cellulose nitrate plastic was included in each run in order to provide information regarding the relative intensity of the ultraviolet light from the six different S-1 bulbs employed. The results of these tests were compared with the data obtained after exposure of the same plastics for 1 yr. on the roof. Excellent qualitative and good quantitative agreement between the laboratory and outdoor tests was observed.

COMPARISON OF THE EFFICACY OF VARIOUS LIGHT SOURCES USED IN CONJUNCTION WITH THE FOG CHAMBER IN ACCELERATED AGING TESTS

Although the Sunlamp with S-1 bulb provides a relatively inexpensive source of ultraviolet light, both in initial outlay and operating cost, and can be run for long periods without an operator's attention, many laboratories with other types of ultraviolet light equipment may wish to know whether combination of the fog chamber feature with exposure in their apparatus would yield results comparable with outdoor exposure. A series of tests was, therefore, made with a representative group of transparent plastics in which the light sources were (1) an S-1 bulb, (2) an S-4 bulb, (3) an H-4 bulb, (4) an H-4 bulb with Nonex glass envelope removed, (5) a quartz-tube mercury-arc lamp with Corex D glass filter, (6) an Eveready Sunshine-carbon arc with Corex D glass filter, (7) a Fade-Ometer carbon arc inclosed in a Corex D glass globe, and (8) a Weather-Ometer carbon arc inclosed in a Corex D glass globe. The radiation and wetting cycles that were employed, each 24 hr., are as follows: 2 hr. in the fog chamber, 2 hr. in the irradiation cabinet, 2 hr. in the fog chamber, and the remainder of the day (slightly under 18 hr. because of the time required to shift the samples) in the irradiation cabinet. The face of each specimen which was exposed directly to the light was turned down in the fog chamber and, in addition, was wiped

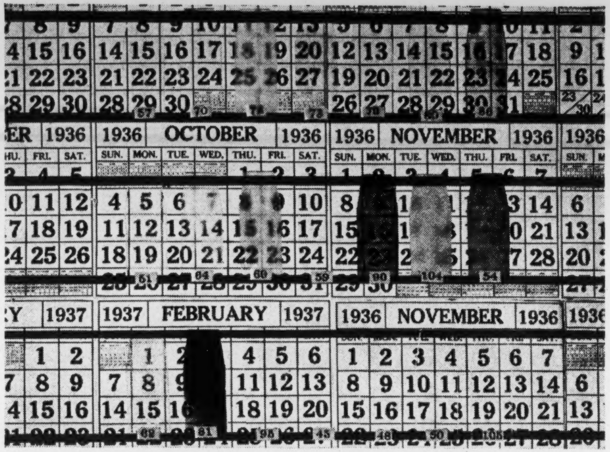

FIG. 8.—S-1 Bulb - Fog Chamber Test.

Specimen	Material
No. 57	Cellulose acetate B57
No. 70	Cellulose acetate B76
No. 72	Cellulose acetate B79
No. 73	Cellulose acetate B80
No. 79	Cellulose acetate-butyrate B86
No. 80	Cellulose acetate B87
No. 86	Cellulose acetate C17
No. 51	Cellulose acetate A6
No. 64	Cellulose acetate A17
No. 69	Cellulose acetate A22
No. 59	Cellulose acetate-butyrate BB1
No. 90	Ethylcellulose I3
No. 104	Ethylcellulose G3
No. 54	Cellulose nitrate W1
No. 62	Vinyl butyral resin N3
No. 81	Vinyl chloride-acetate resin L1
No. 95	Vinyl chloride-acetate resin L7
No. 45	Methyl methacrylate resin K20
No. 48	Methyl methacrylate resin K23
No. 50	Methyl methacrylate resin J11
No. 105	Modified methacrylate resin Y1

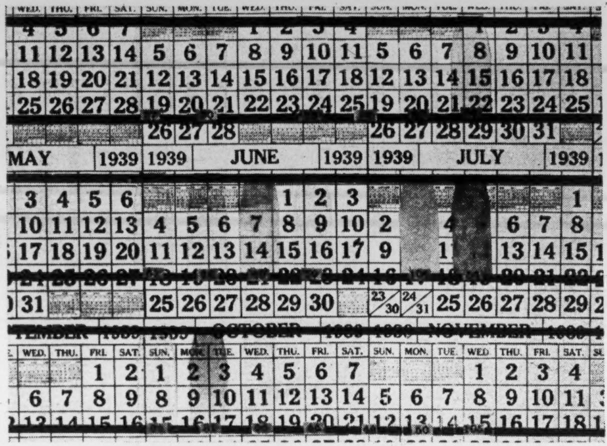

FIG. 9.—S-4 Bulb - Fog Chamber Test.
See Fig. 8 for identification of specimens.

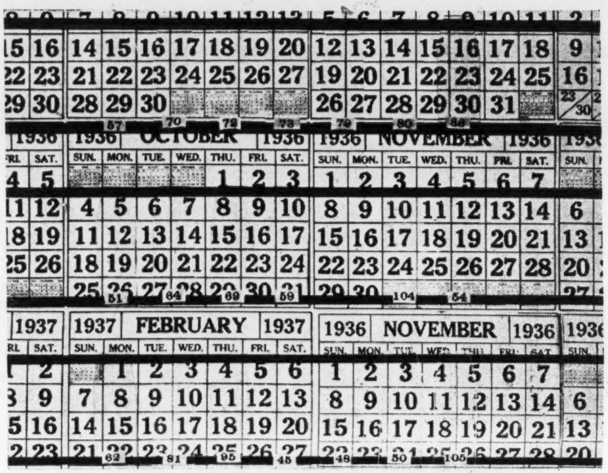

FIG. 10.—H-4 Bulb - Fog Chamber Test.
See Fig. 8 for identification of specimens.

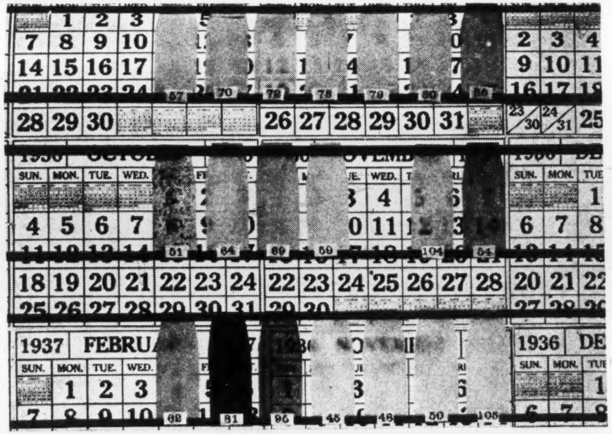

Fig. 11.—H-4 Bulb - Fog Chamber Test Without Glass Envelope on the Bulb.

See Fig. 8 for identification of specimens.

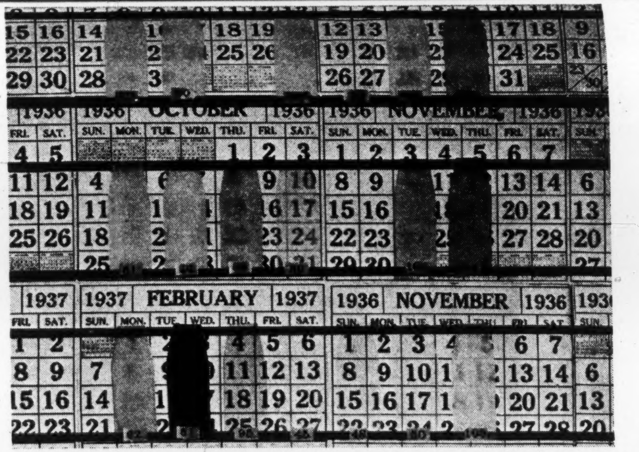

Fig. 12.—Alpine Quartz-Tube Mercury-Arc Lamp - Fog Chamber Test.

See Fig. 8 for identification of specimens.

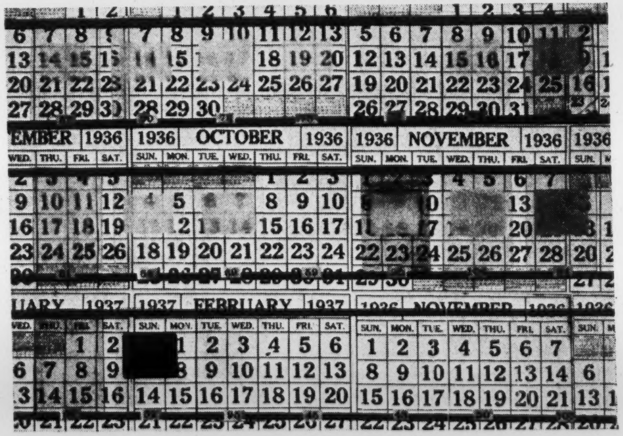

Fig. 13.—Eveready Accelerated Testing Unit - Fog Chamber Test.

See Fig. 8 for identification of specimens.

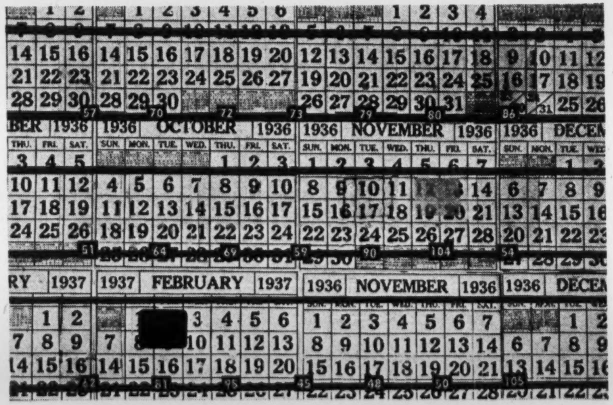

Fig. 14.—Fade-Ometer - Fog Chamber Test.

See Fig. 8 for identification of specimens.

off daily with lens paper wet with distilled water. The results of light transmission and haze measurements in these tests are presented in Table II. Figures 8 to 15, inclusive, show the condition of the samples after 500 hr. of light exposure, except that the test with the H-4 bulb with its glass envelope removed was discontinued after 200 hr. because of the extreme breakdown that had then taken place. Figure 16 shows the condition of the samples after exposure on the roof for the periods noted in the caption.

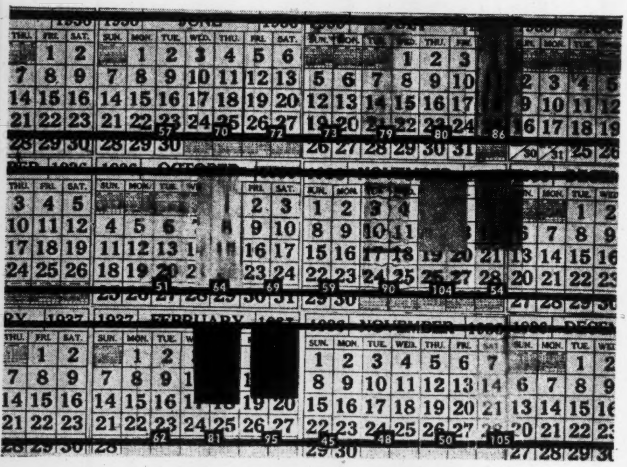

Fig. 15.—Weather-Ometer - Fog Chamber Test.

See Fig. 8 for identification of specimens.

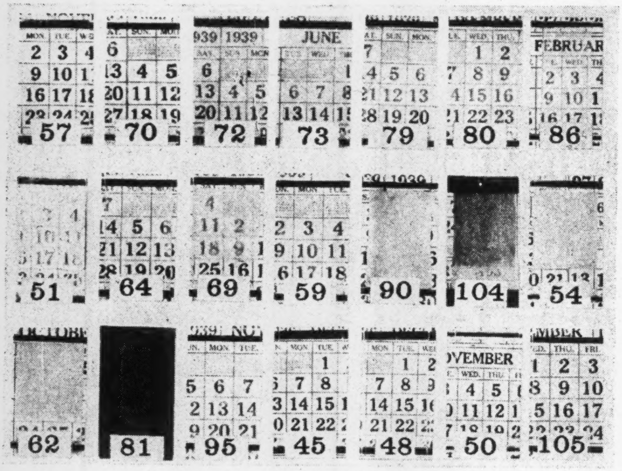

Fig. 16.—Roof Exposure Test.

See Fig. 8 for identification of specimens.

All specimens were exposed outdoors in Washington, D. C., for 1 yr. except as follows: No. 59 exposed for 20 months; No. 90 for 3 months; No. 104 for 6 months; No. 62 for 14 months; No. 95 for 9 months; and No. 105 for 9 months.

An analysis of these various accelerated aging tests with respect to reproducing roof exposure effects is summarized in Table III. The differences between the light transmission and haze values observed after exposure on the roof for one year and after exposure in each of the accelerated aging units were calculated. Then for each specimen the lamps were rated in the order of 1 (best) to 8 (poorest) depending upon their agreement with the roof exposure figure, equal weight being given to plus and minus figures. In case of ties, the total points for the order numbers involved were divided equally

TABLE II.—COMPARISON OF VARIOUS LIGHT SOURCES USED IN COMBINATION WITH FOG CYCLES IN ACCELERATED AGING OF TRANSPARENT PLASTICS.

		Original Specimen, avg., per cent	S-1 Bulb, 500 hr., per cent	S-4 Bulb, 500 hr., per cent	H-4 Bulb, 500 hr., per cent	H-4 Bulb without Glass Envelope, 200 hr., per cent	Alpine Mercury Arc with Corex D Filter, 500 hr., per cent	Eveready Sunshine-Carbon Arc with Corex D Filter, 500 hr., per cent	Fade-Ometer Carbon Arc with Corex D Globe, 500 hr., per cent	Weather-Ometer Carbon Arc with Corex D Globe, 500 hr., per cent	Roof Exposure in Washington, 1 yr., per cent
LIGHT TRANSMISSION OF THE PLASTICS											
Cellulose acetate....	A6	90.0	90.4	91.6	91.0	87.1	46.6	87.9	91.5	91.4	89.6
	A17	89.7	86.5	90.0	90.0	87.0	77.2	82.4	90.5	90.6	90.2
	A22	84.5	82.0	82.8	85.2	80.5	64.6	79.0	87.6	87.8	80.0
	B57	91.6	91.0	91.4	91.0	87.3	71.7	86.7	91.8	91.5	91.5
	B76	91.3	90.3	91.2	91.2	87.0	77.0	86.4	92.0	91.8	92.2
	B79	92.0	84.4	....	92.0	89.6	....	75.2	92.2	92.0	91.5
	B80	91.6	90.0	91.8	92.0	86.4	82.0	88.6	92.1	92.0	91.2
	B86	90.6	90.6	91.2	91.0	87.2	90.2	91.0	90.8	88.7	90.8
	B87	91.9	91.4	92.0	91.9	88.0	82.6	89.2	92.8	92.3	92.0
	C17	86.3	83.5	85.8	86.2	82.1	75.5	80.6	87.2	87.6	87.3
Cellulose acetate-butyrate..........	BB1	89.4	89.6	89.8	89.2	84.5	83.8	89.6	90.0	89.3	89.6
Cellulose nitrate....	W1	87.7	60.5	68.2	86.2	78.2	67.8	56.4	87.8	78.5	38.2
Ethylcellulose.......	G3	87.5	75.8	80.4	87.3	85.6	76.1	75.8	68.3	63.8	
	I3	91.8	77.8	....	....	....	....	85.4	87.9	83.5	
Vinyl butyral.......	N3	71.4	77.0	76.6	72.8	76.0	75.8	82.2	79.6	79.1	9.5
Vinyl chloride-acetate..............	L1	83.8	16.4	69.2	85.1	53.8	9.8	2.0	10.0	0.2	36.0
	L7	83.4	83.0	83.2	82.9	68.7	76.0	81.0	84.1	0.1	
Methyl methacrylate	J11	94.5	94.0	93.8	94.0	91.9	93.4	94.0	94.2	94.0	93.7
	K20	94.2	94.0	93.7	94.0	91.4	92.8	93.5	94.1	93.8	91.6
	K23	94.2	92.4	92.0	93.8	90.6	91.0	92.1	94.2	93.5	90.6
Modified methacrylate..............	Y1	94.1	93.6	93.2	93.8	85.0	77.0	93.0	93.9	82.4	
HAZE VALUES OF THE PLASTICS											
Cellulose acetate....	A6	6.7	10.1	6.4	7.0	32.7	96.6	21.5	7.5	15.4	20.3
	A17	5.2	27.2	5.8	5.3	73.1	70.2	49.5	6.7	29.7	11.3
	A22	5.4	33.9	23.9	5.4	70.0	61.0	44.3	6.4	10.0	28.2
	B57	4.2	5.7	3.8	5.3	72.3	79.9	29.5	6.0	5.0	7.3
	B76	4.0	9.0	4.5	3.5	65.1	57.8	31.7	5.4	5.4	8.0
	B79	3.7	30.6	....	4.6	58.5	....	55.6	6.6	6.7	12.7
	B80	4.3	13.1	6.0	4.7	69.4	66.5	17.8	5.4	4.8	15.1
	B86	3.7	5.6	1.6	3.1	63.3	5.9	3.3	6.5	15.0	9.9
	B87	3.3	7.2	4.6	4.0	73.6	53.8	20.0	5.0	4.4	11.3
	C17	5.4	24.2	10.3	7.0	60.5	58.8	52.9	7.6	38.9	15.1
Cellulose acetate-butyrate..........	BB1	7.8	7.4	7.4	8.3	70.7	32.4	9.4	8.7	9.6	7.9
Cellulose nitrate....	W1	4.8	84.3	60.7	5.0	43.6	80.5	86.7	5.4	5.2	96.9
Ethylcellulose.......	G3	12.2	56.4	80.5	13.2	51.8	68.5	63.3	59.2	91.8	
	I3	5.1	7.2	....	....	....	....	58.0	7.4	18.4	
Vinyl butyral.......	N3	5.2	22.9	7.3	8.1	60.3	69.2	7.8	6.8	16.4	99.0
Vinyl chloride-acetate..............	L1	9.3	[a]	7.7	11.9	25.8	[a]	[a]	[a]	[a]	26.1
	L7	6.9	7.0	9.0	8.1	18.5	8.6	9.8	7.7	[a]	
Methyl methacrylate	J11	1.7	2.1	3.2	2.1	53.9	6.4	3.3	2.2	5.2	1.8
	K20	2.3	2.7	3.1	3.3	39.8	6.0	3.5	3.5	3.6	3.1
	K23	2.1	1.8	3.0	3.3	34.9	6.4	3.6	3.4	3.4	2.7
Modified methacrylate..............	Y1	2.7	4.9	5.2	3.9	79.2	50.5	3.6	4.8	24.8	

[a] Haze could not be measured accurately on this sample because of low light transmission attributable to discoloration rather than to clouding or crazing.

between the lamps which were tied. These points were then added and the lamp with the lowest numerical sum was rated 1 in Table III, the next lowest 2, and so forth. In the visual examination, the specimens were evaluated as (1) transparent, (2) semi-transparent, (3) translucent, (4) semi-opaque, and (5) opaque. The agreement between the roof exposure test and the accelerated tests was rated by the same system employed for the light transmission and haze measurements. It is readily apparent from the totals shown in Table III that the S-1 Sunlamp proved to be the most suitable ultraviolet light source in these tests and that the H-4 lamp without its glass envelope was the poorest.

TABLE III.—COMPARATIVE RATINGS OF THE VARIOUS ACCELERATED AGING TESTS WITH RESPECT TO REPRODUCING ROOF EXPOSURE EFFECTS.

(1 is best, 8 is poorest).

Basis of Rating[a]	S-1 Bulb	S-4 Bulb	H-4 Bulb	H-4 Bulb Without Envelope	Alpine Mercury Arc	Eveready Sunshine-Carbon Arc	Fade-Ometer	Weather-Ometer
Light transmission....	2	1	3	7	8	5.5	5.5	4
Haze values..........	1	3	4	8	7	5	2	6
Visual examination...	1	2	6	8	7	5	3	4
Total points.......	4	6	13	23	22	15.5	10.5	14

[a] Specimens A6, A17, A22, B57, B76, B80, B86, B87, C17, BB1, W1, N3, L1, J11, K20, and K23 were used for obtaining the ratings shown in this table except that L1 was not included in rating the haze measurements.

In considering Table III it must be kept in mind that the time of exposure (500 hr.) was originally selected on the basis of preliminary tests with the S-1 lamp. Other conditions of test with respect to period of exposure, temperature, wetting, and light filters could possibly be found for the other lamps which would bring them into closer agreement with the roof tests with respect to light transmission, haze, or visual examination. It is also obvious that the system of rating employed does not give any weight to the closeness of the agreement with the roof exposure test, and that there may be a greater gap between one test and its neighbor in the order of ranking than is indicated by the single point.

A study of Figs. 8 to 15 reveals a number of outstanding facts concerning both the materials and the various tests. Looking at the specimens in Fig. 8 across the rows from top to bottom, the first ten specimens are cellulose acetate plastics except No. 79. A tremendous difference in the resistance of these cellulose acetate samples to the S-1 Sunlamp - fog chamber aging test is noted. Specimen No. 79 is a cellulose acetate-butyrate material and is particularly outstanding among the cellulose plastics in its resistance to ultraviolet light, as will be more evident in some of the later figures. Specimen No. 59 is also a cellulose acetate-butyrate and is quite resistant to weathering. Specimens 90 and 104 are ethylcellulose plastics, which are rapidly deteriorated when exposed on the roof. It is evident in the photograph that specimen 90, which originally was colorless, is considerably discolored, but the small amount of crazing on this specimen does not show clearly. On the roof this specimen is badly discolored in three months and is also badly crazed, more closely resembling specimen 104 than specimen 90 in Fig. 8 in this respect. The greatest discrepancy between roof exposure results and the S-1 accelerated test observed in all of the experiments that we have made, occurred with this particular specimen of ethylcellulose. Specimen 54 is the cellulose nitrate plastic which was used throughout our tests with the S-1 lamp to check the comparative intensities of the light emitted by the various bulbs. Specimen 62 is a vinyl butyral plastic which obviously is not resistant to the combined effects of light and moisture. Of course, this may be attributable to the specific plasticizer in the composition rather than any inherent instability of that particular type of resin, as was also the case for some of the cellulose acetate compositions previously discussed. Specimens Nos. 81 and 95 present an amazing contrast in the behavior of two copolymer vinyl chloride-acetate plastics. Specimen No. 81 is the old type of sheet material, originally received by us in 1935, whereas specimen No. 95 is a modified composition of this same type of plastic, received in 1939. The latter material is practically unaffected after the 500-hr. test in the accelerated aging apparatus, whereas the former product is nearly opaque from discoloration. This difference in behavior of these two samples has been duplicated in roof exposure tests on these specimens. The remainder of the specimens in the bottom row are methyl methacrylate plastics which are outstanding in weathering resistance, both in these accelerated tests and in roof exposures.

The condition of the set of samples exposed to ultraviolet light from the S-4 lamp in conjunction with the regular fog chamber treatments is shown in Fig. 9. It will be noted that the breakdown of the samples has not proceeded quite so far in this experiment as was observed with the S-1 lamp (Fig. 8), but that it is appreciably greater than was observed for the H-4 bulb (Fig. 10), which utilizes the same capillary mercury-arc unit as the light source.

It is apparent from inspection of Fig. 10 that the H-4 bulb is a comparatively weak source of ultraviolet light. It will be recalled that the Nonex glass used on this bulb cuts off the ultraviolet light at 3000 Å, whereas the glass on the S-1, S-4, and Corex D globe and plates used in the other tests is rated as transmitting ultraviolet light rays down to 2800 Å. When this glass envelope is removed, the short ultraviolet light rays have an extremely rapid and severe deteriorating effect on the plastics with the exception of the copolymer vinyl resins. The effects noted in Fig. 11 are not at all characteristic of the behavior of the plastics on the roof.

The plastics exposed to the Alpine mercury-arc lamp with a Corex D glass filter interposed between the quartz tube and the specimens were also severely deteriorated as is evident in Fig. 12. This can be attributed to the failure of the filter to absorb all of the short ultraviolet light rays from this intense light source. The great superiority of cellulose acetate butyrate specimen No. 79 compared with the other cellulose plastics in resistance to weathering is especially outstanding in this test. Specimen 105 is a sheet of an experimental methacrylate resin which proved to be inferior to the usual commercial grade of methyl methacrylate plastic in these accelerated aging tests as well as on roof exposure.

Figure 13 shows the condition of the plastics after 500 hr. of exposure to the light from a Sunshine carbon arc. The types of breakdown of the specimens are very similar to those observed in the S-1 test and roof exposure. For most of the specimens the deterioration has advanced further in this test than in the S-1 test. Specimens Nos. 79, 59, and 95, as well as all of the methyl methacrylate specimens are particularly noteworthy with respect to their resistance to deterioration in this test. The comparatively negligible breakdown of most of the plastics when exposed in the Fade-Ometer is evident from Fig. 14. An outstanding feature of the Weather-Ometer test, as shown in Fig. 15, is the marked discoloration of both copolymer vinyl resins, specimens Nos. 81 and 95. This is believed to be due to the higher temperature reached by the specimens in this test, because of both a lack of satisfactory cooling facilities and immediate contact of the back of the samples with the metal holders. This anomalous effect is also evident in the condition of specimen No. 79 and in the generally fused appearance of many of the specimens, particularly in the lower portion which was protected from the direct rays of light.

CONCLUSION

The following accelerated aging procedure and apparatus are recommended for rapidly evaluating the weathering resistance of transparent plastics. The apparatus consists of four parts: (1) a Sunlamp, model BM6, equipped with a new S-1 bulb for each 500-hr. test; (2) a phonograph turntable, operating at 33 rpm., on which is mounted an aluminum disk 20 in. in diameter and 0.1 in. thick; (3) a cabinet approximately 3 ft. high, 2 ft. wide, and 2 ft. deep, open on one side; (4) a fog chamber which consists of a closed shallow box fitted with a spraying unit and a baffle to prevent the spray from impinging directly on the plastics. The test specimens which are 4 in. long and 1.5 in. wide with a tapered end are supported about $\frac{3}{16}$ in. above the surface of the aluminum disk by means of two sets of 21 bolts attached to the disk equidistantly on circles $11\frac{3}{4}$ and $15\frac{3}{4}$ in. in diameter, respectively. The disk is centered under the S-1 bulb so that the plane of the specimens is 12 in. from the bottom of the bulb. The radiation and wetting cycles that are employed each 24 hr. are as follows: 2 hr. in the fog chamber, 2 hr. in the irradiation cabinet, 2 hr. in the fog chamber, and the remainder of the day (slightly under 18 hr. because of the time required to shift the samples) in the irradiation cabinet. The light transmission and haze values of the transparent plastics are measured at the conclusion of 500 hr. of exposure to the ultraviolet light, and the specimens are examined for evidences of crazing, fusing, blooming, discoloration, and warping. This test is approximately equivalent to exposure for one year in Washington, D. C., on a rack facing south and inclined at an angle of 45 deg. to the horizontal.

DISCUSSION

MR. M. L. MACHT[1] (*presented in written form*).—The procedure for accelerated weathering of transparent plastics, involving alternate exposure to a misty atmosphere and to light from an S-1 Sunlamp bulb, developed by Messrs. Kline, Crouse, and Axilrod, has been used in our research laboratories for the past year. Cellulose derivatives as well as methyl methacrylate have been subjected to this test with results that approximate those obtained on outdoor weathering. The effect on methacrylate plastics is somewhat less pronounced than that of outdoor Florida exposure, as contrasted with the effect on cellulose derivative plastics which is more pronounced than that of Florida exposure.

Comparative testing of identical materials by this accelerated method as compared to actual exposure at Arlington, N. J., and at Hialeah, Fla., is at present in course. While these tests are not as yet complete, it is known that 500-hr. exposure to the accelerated weathering cycle is considerably more effective than 1-yr. exposure at Arlington. In at least one case, 500 hr. has shown up an experimental methacrylate composition which failed in one year in Florida.

MR. H. A. NELSON.[2]—I should like to ask what the authors know about the effects of infrared and the longer wave lengths. Do these rays build up the temperature in plastic materials and show specific deteriorating effects?

MR. G. M. KLINE.[3]—Yes, the plastics reach a temperature of about 15 to 20 deg. higher than that of the surrounding air, so that presumably the infrared rays are building up a certain amount of heat in the plastics themselves. We have to be careful in our work to distinguish between the air temperature and the actual temperature of the plastic. It is necessary to actually bore a hole longitudinally in the plastic and by means of a thermocouple establish that the temperature is below the 60 C. limit, which will lead to more pronounced thermal effects than to actual weathering effects.

MR. PHILIP SADTLER.[4]—Would a closed Weather-Ometer with a fan to regulate the temperature so that the temperature would not go above 60 C. give a closer approximation of outside weathering conditions; also would the Fade-Ometer with the mist spray instead of the rain spray give results nearer the roof conditions?

MR. KLINE.—Any apparatus which would keep the temperature of the samples at 55 to 60 C. would be satisfactory, from the thermal standpoint. I am thinking now of the difference, for example, between the Fade-Ometer and the Weather-Ometer, in which the one has the fan and the temperature control and the other is open and the temperature does go up appreciably. As far as the mist is concerned I do not see that it makes a great deal of difference whether you obtained that mist in the apparatus with the light shut off or whether you transfer the plastics to a separate chamber for the mist. As far as the light source is concerned, I think that it is quite necessary to be sure that the light source that you are using does very closely simulate the spectrum that you get from sunlight itself.

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[3] Chief, Organic Plastics Section, National Bureau of Standards, Washington, D. C.

[4] Vice-President, Samuel P. Sadtler and Son, Inc., Philadelphia, Pa.

MR. SADTLER.—Why is the light turned off when the mist is put on?

MR. KLINE.—For the reason that the heat from the lamp would result in drying off the specimen practically instantaneously. We have run an experiment in which we simply took the plastic out from under the light, sprayed it and put it right back. This does not produce the same type of break-down that occurs when the moisture is in contact with the sample for an appreciable period, say, one or two hours, and then the sample is put under the light and allowed to dry. That is, if you compare this treatment with your roof conditions, dew does stay on your sample for a very appreciable time and then dries off. You will probably get diffusion of decomposition products from the interior of your plastic, that will not take place if you simply sprayed the surface and it dried off practically immediately.

I might say in connection with Mr. Macht's discussion that we have completed exposures in Washington, Florida and Panama ourselves, and we have had the same plastics exposed in the accelerated weathering equipment which we have described in this paper. These experiments will be the subject of another paper. This particular paper was concerned merely with the discussion of the new weathering test and comparison of it with tests employing other light sources.

Since this paper was prepared and presented, our accelerated weathering test has been modified to the extent of removing the turntable and Sunlamp from the cabinet to a table in a room kept at 25 C. by air-conditioning equipment. This has enabled us to decrease the distance between the bulb and specimens to 6 in. and still maintain a sample temperature of about 60 C. By this modification we are able to produce in 200 hr. of light exposure and twenty 2-hr. fog exposures a breakdown somewhat greater than that which occurred in 500 hr. of light exposure and fifty 2-hr. fog exposures, as described in this paper. Thus, an even more rapid and economical method of evaluating the outdoor weathering characteristics of transparent plastics is provided for use in research and materials specifications.

SORPTION OF WATER BY PLASTICS

By G. M. Kline,[1] A. R. Martin,[1] and W. A. Crouse[1]

Synopsis

A method of measuring the absorption of water by plastics has been prepared by the Society's Committee D-20 on Plastics working in cooperation with Committee D-9 on Electrical Insulating Materials. This method has been published as tentative[2] and replaces the short-time water absorption tests provided for in methods D 48 – 39 for molded insulating materials, D 229 – 39 for sheet and plate materials, D 349 – 39 for laminated round rods, and D 348 – 39 for laminated tubes.

Experimental data obtained in an exploratory investigation at the National Bureau of Standards on the absorption of water by plastics in connection with the preparation of the above test method are summarized in this paper. These data pertain to the effects of various conditioning procedures, times of immersion, and prior history of the specimens on the values observed for water absorption by plastics. The plastics examined were phenol-formaldehyde molded, cast, and paper-base laminated materials, urea-formaldehyde molded and paper-base laminated materials, casein, cellulose nitrate, cellulose acetate, ethylcellulose, polyvinyl chloride-acetate, polyvinyl butyral, polystyrene in molded and cast forms, polymethyl methacrylate, and cold-molded plastics of the bituminous and phenolic types.

Data are also presented on the rates of absorption and desorption of water by these plastics for periods up to two years. The variations in the dimensions of the specimens that are produced by these changes in moisture content are recorded.

There have been numerous reports in the literature during the past three years concerning the sorption of water by various types of plastics and their resulting dimensional changes. The general opinion is that the mechanical and electrical properties and dimensional stability of plastics are definitely affected by water absorbed by them and that low water absorption is of considerable advantage in a plastic material. Another point that is usually stressed is that provided a material has been selected on the basis of mechanical and electrical properties, measurement of water absorption constitutes a simple method of testing to ensure that the desired grade or quality of material is being obtained. Part of the experimental data obtained in an exploratory investigation at the National Bureau of Standards of the absorption of water by plastics in connection with the preparation of a test method is summarized in this paper.

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[1] Chief, Organic Plastics Section, Junior Scientific Aide, and Junior Physicist, respectively, National Bureau of Standards, Washington, D. C.

Materials and Test Methods

The plastics examined were phenol-formaldehyde molded, cast, and paper-base laminated materials, urea-formaldehyde molded and paper-base laminated materials, casein, cellulose nitrate, cellulose acetate, ethylcellulose, polyvinyl chloride-acetate, polyvinyl butyral, polystyrene in molded and cast forms, polymethyl methacrylate, and cold-molded plastics of the bituminous and phenolic types. These plastics were obtained from commercial sources and many of them contained the plasticizers ordinarily present in such compositions. Specimens 1 by 3 in. by the thickness of the samples, which was approximately $\frac{1}{8}$ in., were cut from the sheet materials. The molded specimens were disks 2 in. in diameter and $\frac{1}{8}$ in. thick. The surface areas on these two forms of specimens are approximately equal.

TABLE I.—EFFECT OF TIME OF CONDITIONING AND DURATION OF IMMERSION ON ABSORPTION OF WATER BY PLASTICS.

Material	Thickness, mils	Absorption, 24-hr Immersion, per cent				Absorption, 48-hr. Immersion, per cent			
		After 24 hr. at 50 C.	After 48 hr. at 50 C.	After 2 days at 25 C. and 50% Relative Humidity	After 24 hr. at 105 to 110 C.	After 24 hr. at 50 C.	After 48 hr. at 50 C.	After 2 days at 25 C. and 50% Relative Humidity	After 24 hr. at 105 to 110 C.
Phenol-formaldehyde molded	128	0.34	0.32	0.26	....	0.50	0.47	0.40	....
Phenol-formaldehyde cast	127	0.26	0.25	0.16	0.16	0.37	0.37	0.24	0.22
Phenol-formaldehyde laminated	134	0.49	0.48	0.44	0.39	0.84	0.86	0.82	0.71
Urea-formaldehyde molded	125	0.73	1.02	0.48	....	1.15	1.37	0.76	....
Urea-formaldehyde laminated	132	0.67	0.72	0.38	0.98	0.97	1.04	0.62	1.11
Vinyl chloride-acetate resin	133	0.05	0.05	0.03	0.08	0.07	0.07	0.05	0.11
Vinyl butyral resin	122	1.05	1.01	0.75	....	1.48	1.47	1.12	....
Methyl methacrylate resin	139	0.35	0.33	0.22	0.37	0.46	0.46	0.31	0.53
Styrene resin (molded)	126	0.05	0.05	0.03	....	0.05	0.05	0.03	....
Styrene resin (cast)	156	0.05	0.05	0.03	....	0.06	0.06	0.03	....
Cellulose nitrate	123	0.99	1.00	0.64	....	1.38	1.40	0.91	....
Cellulose acetate	133	2.52	2.61	2.00	2.80	3.54	3.70	2.88	4.00
Ethylcellulose No. 1	185	0.85	0.86	0.57	0.65	1.18	1.20	0.83	1.45
Ethylcellulose No. 2	166	1.07	1.05	0.84	1.24	1.47	1.47	1.16	1.71
Cold-molded phenolic	128	1.03	1.06	0.69	....	1.27	1.23	0.90	....
Cold-molded bituminous	130	0.61	0.58	0.49	....	0.82	0.86	0.84	....
Casein	121	8.46	8.40	8.10	9.95	13.41	12.91	13.49	14.65

The conditioned specimens were placed on edge and wholly immersed in distilled water, which had been freshly boiled and then cooled and maintained at 25 C. Bubbles usually collect on the test specimens when immersed in water that has not been freshly boiled and this causes irregular exposure of the surface to the water. At the desired intervals, the specimens were wiped off with a dry cloth to remove surface water, and weighed immediately. Changes in dimensions were measured with dial gages reading to 0.001 in. and having approximately 400 g. per sq. cm. pressure on the foot. All percentage changes in weights and dimensions are calculated on the basis of the as-received weights and dimensions of the specimens, unless otherwise noted.

Investigation of Conditioning Procedures

The data in Table I pertain both to the effect of various conditioning procedures on the value obtained for water absorption and to the values observed for 24- and 48-hr. immersion. It will be noted that for the specimens of approximately $\frac{1}{8}$ in. thickness conditioned at 50 C. the water absorption value is practically the same for a 24-hr. conditioning period as for the 48-hr. period. The value obtained when the sample is conditioned at 25 C. and 50 per cent relative humidity is lower than that obtained after heating for 24 or 48 hr. at 50 C. without humidity control. This is attributable to the elimination of moisture by heating at 50 C. and the greater rate of regain of this moisture as compared with the subsequent rate of absorption of water. On the other hand, conditioning the samples at 105 to 110 C. for 24 hr. leads to somewhat erratic results as compared with those observed after heating at 50 C. For some materials, notably the phenolic resin products, the water absorption value is lowered, whereas in most instances a considerably higher value is obtained. Some materials, particularly cellulose nitrate, vinyl butyral resin, and styrene resin, deteriorate or soften to such an extent when heated for 24 hr. at 105 to 110 C. that the specimens become unsuitable for the water-absorption test.

TABLE II.—VOLATILE MATTER LOST BY PLASTICS UNDER VARIOUS CONDITIONS.

Material	24 hr. at 50 C.		48 hr. at 50 C.		1 hr. at 105 to 110 C.		24 hr. at 105 to 110 C.	
	mg.	%	mg.	%	mg.	%	mg.	%
Phenol-formaldehyde molded	11	0.12	14	0.14	..	....	..	....
Phenol-formaldehyde cast	27	0.33	39	0.48	70	0.85	294	3.58
Phenol-formaldehyde laminated	16	0.20	28	0.31	28	0.32	121	1.40
Urea-formaldehyde molded	60	0.63	104	1.10	..	....	..	....
Urea-formaldehyde laminated	40	0.43	67	0.69	39	0.41	369	3.91
Vinyl chloride-acetate resin	3	0.03	4	0.04	7	0.08	28	0.32
Vinyl butyral resin	39	0.59	53	0.79	..	....	..	....
Methyl methacrylate resin	19	0.23	23	0.28	28	0.35	58	0.71
Styrene resin (molded)	2	0.03	2	0.03	..	....	..	....
Styrene resin (cast)	2	0.03	2	0.03	7	0.09	15	0.19
Cellulose nitrate	69	0.84	77	0.93	77	0.82	430	5.23
Cellulose acetate	80	0.95	103	1.22	97	1.04	282	3.33
Ethylcellulose No. 1	36	0.36	46	0.45	41	0.41	66	0.65
Ethylcellulose No. 2	46	0.50	48	0.52	55	0.60	85	0.94
Cold-molded phenolic	33	0.24	35	0.26	..	....	..	....
Cold-molded bituminous	16	0.13	24	0.19	..	....	..	....
Casein	105	1.36	142	1.84	129	1.71	511	6.77

TABLE III.—EFFECT OF EXPOSURE OF SAMPLES TO WET OR DRY ATMOSPHERES PRIOR TO MAKING WATER ABSORPTION TEST.

Experiment	Exposure Conditions							Water-Absorption Values[a]					
	72 hr. at 100 per cent relative humidity	72 hr. at 50 C.	72 hr. at room temperature	48 hr. at room temperature	24 hr. at 50 C.	48 hr. in H_2O	24 hr. at 50 C.	Methyl Methacrylate Resin, per cent	Cellulose Acetate, per cent	Ethylcellulose No. 2, per cent	Vinyl Butyral Resin, per cent	Laminated Phenolic Resin, per cent	Laminated Urea Resin, per cent
No. 1	X			X	X	X	X	0.50	4.33	1.69	1.55	1.13	1.07
No. 2	X				X	X	X	0.49	4.22	1.53	1.52	....	....
No. 3		X		X	X	X	X	0.52	4.15	1.62	1.58	0.94	0.87
No. 4		X			X	X	X	0.53	3.96	1.57	1.49	....	....
No. 5			X	X	X	X	X	0.52	4.16	1.60	1.55	0.91	1.07
No. 6			X		X	X	X	0.50	3.94	1.57	1.55	....	....

[a] Based on weight of specimen immediately prior to immersion in water.

The losses in weight of the specimens for the various conditioning procedures are shown in Table II. Heating at 105 to 110 C. for 1 hr. results in losses similar to those produced by exposure at 50 C. for 24 or 48 hr., except for three materials: namely, cast phenolic resin, styrene resin, and vinyl chloride-acetate resin. For these materials the loss in weight is 2 to 3.5 times as great at 105 to 110 C. for 1 hr. as at 50 C. for 24 to 48 hr. Heating at 105 to 110 C. for 24 hr. results in relatively large losses in weight in the case of cellulose nitrate, vinyl butyral, and styrene plastics.

The data in Table III show the effect on water absorption by plastics of an exposure to a wet or dry atmosphere immediately prior to starting the test. The effect of a 2-day interval of exposure at room temperature and humidity after such wetting or drying and before starting the moisture absorption test was also investigated as a possible means of overcoming the consequences of an abnormal past history. The experimental data, not reported in Table III, revealed that 24-hr. heating at 50 C. was sufficient to remove the water taken up by the samples when exposed in a saturated atmosphere for 3 days. Also, the total amount of water driven off on allowing the samples, which had been exposed in a saturated atmosphere for 3 days, to stand in the air 2 days followed by 24 hr. heating at 50 C. was approximately the same as that removed by heating the samples for 24 hr. at 50 C. immediately following the 3 days' exposure to 100 per cent relative humidity. The values presented in Table III for water absorption after these various treatments show very good agreement in most instances. It is believed to be quite conclusive evidence that a 24-hr. conditioning period at 50 C. preparatory to making water-absorption tests is sufficient for samples $\frac{1}{8}$ in. thick.

The effect of eliminating the preliminary conditioning at 50 C. and using the conditioned weight obtained after immersion as a basis for calculating the percentage moisture absorption is shown in Table IV. It will be noted that if the difference between the weight of the sample after immersion and its original weight is taken as the amount of water absorbed, the values (method B) for percentage absorption are lower than those obtained by prior conditioning (method A) because under condition B the moisture content of the samples as received retards the absorption of further quantities of water. On the other hand, if the difference between the weight of the sample after immersion and its weight after subsequent conditioning at 50 C. for 48 hr. is taken as the amount of water absorbed, the values (method C) are higher than those obtained by the usual method A because under condition C a portion of the initial moisture content of the material is removed by the conditioning at 50 C. along with the water absorbed during immersion.

TABLE IV.—EFFECT OF IMMEDIATE IMMERSION OF SPECIMEN ON WATER-ABSORPTION VALUE.

Material	Water Absorption, per cent		
	Method A	Method B	Method C
Methyl methacrylate resin	0.46	0.37	0.62
Laminated phenolic resin	0.87	0.79	1.13
Laminated urea resin	1.06	0.38	1.06
Ethylcellulose No. 2	1.48	1.17	1.72
Vinyl butyral resin	1.61	1.14	1.98
Cellulose acetate	4.24	2.74	4.92
Casein	13.15	12.36	14.15

Method A.—Exposed for 48 hr. at 50 C. prior to immersion for 48 hr. in water at 25 C. and conditioned for 48 hr. at 50 C. after immersion.

$$\%\ \text{absorption} = \frac{\text{wet wt.} - \text{conditioned wt.}}{\text{conditioned wt.}} \times 100$$

Method B.—Immersed immediately and conditioned for 48 hr. at 50 C. after immersion.

$$\%\ \text{absorption} = \frac{\text{wet wt.} - \text{original wt.}}{\text{conditioned wt.}} \times 100$$

Method C.—Immersed immediately and conditioned for 48 hr. at 50 C. after immersion.

$$\%\ \text{absorption} = \frac{\text{wet wt.} - \text{conditioned wt.}}{\text{conditioned wt.}} \times 100$$

COMPARISON OF VARIOUS METHODS OF REPORTING WATER ABSORPTION

The plastics investigated are arranged in Table V in the order of increasing values for water absorption per unit surface area when conditioned at 50 C. for 48 hr. and immersed in water for 48 hr. Other values given in the table are the weight of water taken up by the specimens, the percentage absorption based on the as-received weight of the specimens, and the absorption per unit volume of the original specimen. If the water absorption per unit of surface area is assumed to line the materials up in the proper order, then the cold-molded and vinyl butyral specimens appear to be out of place in the column giving the percentage changes in weight. This can be accounted for by the high specific gravity of the cold-molded specimens and the low specific gravity of the vinyl butyral resin. The ethylcellulose materials are out of place in the column showing the amount of water absorbed per unit volume because these specimens were considerably thicker than those of the other materials and hence give lower values when the results are calculated per unit volume. The only significant displacement noted in values obtained for 24 hr. conditioning and 24 hr. immersion in water, but not shown in Table V, was in the position of the urea-formaldehyde molded material. The urea-formaldehyde molded and laminated samples were found to be quite erratic in their behavior in these tests, but in general the position of the molded material will be immediately following the laminated material.

TABLE V.—COMPARISON OF VARIOUS METHODS OF REPORTING WATER ABSORPTION.
All specimens were conditioned at 50 C. for 48 hr. and immersed in water at 25 C. for 48 hr.

Material	Thickness, mils	Specific Gravity	Water Absorption Based on:			
			Weight of Water Absorbed, mg.	Conditioned Weight of Specimen, per cent	Surface Area of Specimen, g. per sq. m.	Volume of Specimen, g. per cu. dm.
1. Styrene resin molded	126	1.03	3.5	0.05	0.8	0.6
2. Styrene resin cast	156	1.03	4.8	0.06	1.0	0.6
3. Vinyl chloride-acetate resin	133	1.35	6.4	0.07	1.4	1.0
4. Phenol-formaldehyde resin cast	127	1.29	30.3	0.37	6.7	4.9
5. Methyl methacrylate resin	139	1.16	37.2	0.46	8.1	5.4
6. Phenol-formaldehyde resin molded	128	1.40	43.2	0.47	9.4	6.9
7. Phenol-formaldehyde resin laminated	134	1.33	75.9	0.87	16.6	11.6
8. Vinyl butyral resin	122	1.09	96.8	1.48	21.5	16.1
9. Urea-formaldehyde resin laminated	132	1.46	99.8	1.06	22.1	15.4
10. Cold-molded bituminous	130	1.87	111.2	0.87	24.3	17.4
11. Ethylcellulose No. 1	185	1.10	121.3	1.20	25.2	13.4
12. Cellulose nitrate	123	1.36	114.9	1.40	25.5	19.0
13. Urea-formaldehyde resin molded	125	1.47	130.1	1.39	28.5	21.2
14. Ethylcellulose No. 2	166	1.12	136.2	1.48	28.8	16.7
15. Cold-molded phenolic	128	2.09	169.3	1.24	37.0	26.9
16. Cellulose acetate	133	1.28	313.0	3.75	68.6	47.9
17. Casein	121	1.25	997.0	13.15	221.8	167.6

ABSORPTION OF WATER OVER LONG PERIODS

Data on the absorption of water by plastics during long-continued immersion are presented in Table VI and Fig. 1. Four specimens of each plastic were submitted to different conditioning procedures and were then placed in water. It will be observed that the prior conditioning does affect appreciably the amount of moisture absorbed by the plastic. However, when the gains or

TABLE VI.—MOISTURE ABSORPTION BY PLASTICS DURING 8 WEEKS' IMMERSION IN WATER AFTER VARIOUS CONDITIONING PROCEDURES.

Material	Gain in 48 hr. at 25 C. and 50% Relative Humidity, per cent	Loss in 24 hr. at 50 C., per cent	Loss in 48 hr. at 50 C., per cent	Gain in 48 hr. at 50 C and 90% Relative Humidity, per cent	Loss in 48 hr. at 50 C., per cent	Gain During Immersion in Water, per cent				Net Gain in Weight, per cent
						24 hr.	48 hr.	7 days	8 weeks	
Phenol-formaldehyde molded	0.02	....	....	....	....	0.25	0.40	0.91	4.69	4.71
	....	0.12	....	....	....	0.34	0.50	1.07	4.99	4.87
	....	....	0.16	....	....	0.32	0.49	1.04	4.82	4.66
	....	....	....	0.45	0.44	0.38	0.51	0.96	4.40	4.41
Phenol-formaldehyde cast	−0.02	....	....	....	....	0.16	0.25	0.54	1.87	1.85
	....	0.32	....	....	....	0.24	0.37	0.74	2.16	1.84
	....	....	0.43	....	....	0.25	0.36	0.78	2.34	1.91
	....	....	....	0.66	0.82	0.31	0.47	0.86	2.07	1.91
Phenol-formaldehyde laminated	0.06	....	....	....	....	0.44	0.84	2.24	6.38	6.42
	....	0.19	....	....	....	0.49	0.83	....	....	....
	....	....	0.31	....	....	0.48	0.86	2.26	6.48	6.17
	....	....	....	0.29	0.55	0.55	0.93	2.35	6.42	6.16
Urea-formaldehyde molded	0.10	....	....	....	....	0.49	0.79	1.98	5.02	5.12
	....	0.59	....	....	....	0.75	1.07	2.20	5.45	4.86
	....	....	1.15	....	....	1.12	1.50	3.21	6.88	5.73
	....	....	....	1.32	1.90	1.20	1.71	2.95	4.66	4.08
Urea-formaldehyde laminated	0.07	....	....	....	....	0.40	0.65	1.44	3.50	3.57
	....	0.44	....	....	....	0.70	1.01	1.93	4.35	3.91
	....	....	0.75	....	....	0.77	1.07	1.96	4.40	3.65
	....	....	....	1.54	2.02	1.30	1.73	2.64	3.82	3.34
Vinyl chloride-acetate resin	0.01	....	....	....	....	0.04	0.05	0.08	0.13	0.14
	....	0.03	....	....	....	0.05	0.06	0.11	0.16	0.13
	....	....	0.05	....	....	0.05	0.07	0.12	0.17	0.12
	....	....	....	0.09	0.11	0.05	0.06	0.10	0.14	0.12
Vinyl butyral resin	0.04	....	....	....	....	0.75	1.12	2.24	5.83	5.87
	....	0.60	....	....	....	1.07	1.52	2.80	6.54	5.94
	....	....	0.82	....	....	1.02	1.54	2.87	6.77	5.95
	....	....	....	1.59	2.06	0.99	1.39	2.55	6.08	5.61
Methyl methacrylate resin	0.05	....	....	....	....	0.21	0.31	0.60	1.17	1.22
	....	0.23	....	....	....	0.33	0.45	0.80	1.43	1.20
	....	....	0.29	....	....	0.32	0.45	0.83	1.48	1.19
	....	....	....	0.45	0.64	0.33	0.46	0.83	1.39	1.20
Styrene resin molded	0.01	....	....	....	....	0.03	0.03	0.03	0.03	0.04
	....	0.03	....	....	....	0.05	0.05	0.05	0.05	0.02
	....	....	0.04	....	....	0.05	0.05	0.05	0.05	0.01
	....	....	....	0.04	0.06	0.04	0.04	0.04	0.05	0.03
Styrene resin cast	0.00	....	....	....	....	0.03	0.03	0.04	0.04	0.04
	....	0.03	....	....	....	0.05	0.06	0.06	0.06	0.03
	....	....	0.02	....	....	0.05	0.06	0.06	0.06	0.04
	....	....	....	0.04	0.06	0.04	0.06	0.06	0.06	0.04
Cellulose nitrate	−0.10	....	....	....	....	0.64	0.91	1.43	1.27	1.3[a]
	....	0.83	....	....	....	0.98	1.37	2.07	1.97	1.2[a]
	....	....	0.92	....	....	0.97	1.38	2.12	2.04	1.2[a]
	....	....	....	0.77	1.61	0.94	1.32	1.97	1.83	1.1[a]
Cellulose acetate	0.17	....	....	....	....	2.02	2.90	4.16	3.09	4.3[a]
	....	0.93	....	....	....	2.52	3.56	....	....	....
	....	....	1.22	....	....	2.61	3.70	5.23	4.45	4.0[a]
	....	....	....	2.00	3.17	2.48	3.43	4.83	4.02	3.7[a]
Ethylcellulose No. 1	0.11	....	....	....	....	0.59	0.86	1.50	2.57	2.68
	....	0.32	....	....	....	0.83	1.16	1.90	3.03	2.71
	....	....	0.46	....	....	0.85	1.18	1.91	3.02	2.56
	....	....	....	1.23	1.50	0.81	1.13	1.87	2.79	2.52
Ethylcellulose No. 2	0.02	....	....	....	....	0.82	1.15	1.99	3.19	3.21
	....	0.50	....	....	....	1.08	1.50	2.42	3.62	3.12
	....	....	0.51	....	....	1.05	1.50	2.44	3.63	3.12
	....	....	....	1.27	1.76	1.05	1.45	2.36	3.47	2.98
Cold-molded phenolic	0.00	....	....	....	....	0.61	0.84	1.21	2.06	(2.06)
	....	0.23	....	....	....	0.86	1.08	1.66	2.88	2.65
	....	....	0.25	....	....	0.98	1.30	1.81	3.06	2.81
	....	....	....	0.29	0.53	1.10	1.27	1.82	2.86	2.62

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[a] Approximate values only; solution of water-soluble constituents has masked the actual increase in weight of the sample attributable to the absorption of water.

TABLE VI.—*Continued.*

Material	Gain in 48 hr. at 25 C. and 50% Relative Humidity, per cent	Loss in 24 hr. at 50 C., per cent	Loss in 48 hr. at 50 C., per cent	Gain in 48 hr. at 50 C. and 90% Relative Humidity, per cent	Loss in 48 hr. at 50 C., per cent	Gain During Immersion in Water, per cent				Net Gain in Weight, per cent
						24 hr.	48 hr.	7 days	8 weeks	
Cold-molded bituminous	0.06	....	....	....	....	0.54	0.84	1.83	4.64	4.70
	....	0.15	....	....	....	0.59	0.88	1.80	4.68	4.53
	....	....	0.20	....	....	0.63	0.93	1.80	4.74	4.54
	....	....	....	0.63	0.61	0.71	1.01	1.95	4.77	4.79
Casein	0.13	....	....	....	....	8.68	14.25	28.18	35.08	35.21
	....	1.39	....	....	....	7.50	11.94	29.17	37.31	35.92
	....	....	1.89	....	....	8.28	12.82	30.06	37.26	35.37
	....	....	....	4.65	5.58	9.52	14.32	29.70	36.08	35.15

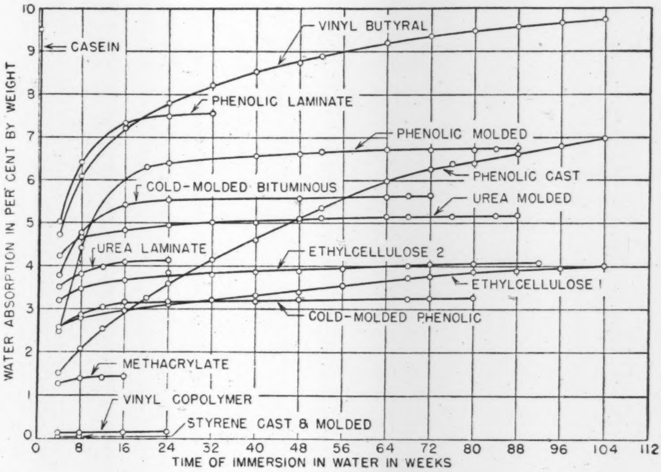

Fig. 1.—Absorption of Water by Plastics.

losses in the various conditioning treatments are taken into consideration, there is very good agreement between the net amounts of moisture absorbed by the four specimens of each plastic, as shown in the last column of Table VI. Again it will be noted that the urea-formaldehyde plastics show the least uniform behavior in these tests.

The specimens of cellulose nitrate and cellulose acetate show a decrease in weight between the 7-day and 8-week periods. This is caused by the dissolution of water-soluble constituents of the plastic which masks the actual increase in water content. This dissolving action is, of course, actually taking place from the instant of immersion of the specimen and is overbalanced during the early stages by the water-absorptive capacity of the plastic. In order to avoid obtaining a false figure for the amount of water absorbed by plastics containing water-soluble ingredients, a requirement has been inserted in the A.S.T.M. tentative method[2] for reconditioning the specimen after the period of immersion. In the tests reported in Table I, in which the specimens were heated at 50 C. for 48 hr. and immersed in water for 48 hr. at 25 C., reconditioning at 50 C. for 48 hr. revealed that four materials had lost some soluble matter: namely, phenol-formaldehyde cast resin 0.10 per cent, cellulose nitrate 0.14 per cent, cellulose acetate 0.49 per cent, and vinyl butyral resin 0.15 per cent. These figures should, therefore, be added to the 48-hr. values in Table I to obtain a corrected figure for weight increase because of water absorption. The loss of soluble matter during the 24-hr. immersion period specified in the A.S.T.M. tentative method[2] would be somewhat less than the values reported above for a 48-hr. period.

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[2] Tentative Method of Test for Water Absorption of Plastics (D 570 - 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 292.

TABLE VII.—MOISTURE DESORPTION BY PLASTICS.

Material	Gain in 48 hr. at 25 C. and 50% Relative Humidity, per cent	Loss in 48 hr. at 50 C., per cent	Gain in 48 hr. in Water at 25 C., per cent	Loss in 24 hr. at 25 C. and 50% Relative Humidity, per cent	Loss Over P_2O_5, per cent[a]				Net Loss in Weight, per cent
					24 hr.	48 hr.	7 days	8 weeks	
Phenol-formaldehyde cast	−0.04	....	....	....	0.10	....	0.30	0.94	0.98
	....	0.50	0.37	0.12	0.30	0.37	0.54	1.12	1.25
Phenol-formaldehyde laminated	0.05	....	....	....	0.13	0.21	0.37	0.86	0.81
Urea-formaldehyde laminated	....	0.65	1.00	0.33	0.90	1.08	1.53	2.78	2.43
Vinyl chloride-acetate resin	0.01	....	....	....	0.04	0.05	0.09	0.11	0.10
	....	0.04	0.08	0.03	0.06	0.08	0.10	0.11	0.07
Vinyl butyral resin	0.04	....	....	....	0.38	....	0.90	1.58	1.54
	....	0.78	1.42	0.58[b]	1.04	1.19	1.60	2.20	1.56
Methyl methacrylate resin	0.04	....	....	....	0.19	0.25	0.43	0.60	0.56
Styrene resin cast	0.00	....	....	....	0.04	....	0.04	0.04	0.04
	....	0.02	0.06	0.03[b]	0.07	0.08	0.08	0.08	0.04
Cellulose nitrate	−0.12	....	....	....	0.54	....	1.08	1.30	1.42
	....	0.93	1.41	0.43	1.09	1.34	1.77	2.00	1.52
Cellulose acetate	0.24	....	....	....	0.76	1.16	1.56	1.75	1.51
Ethylcellulose No. 1	0.13	....	....	....	0.52	0.60	0.72	0.74	0.61
	....	0.44	1.15	0.39	0.85	1.02	1.29	1.34	0.63
Ethylcellulose No. 2	0.04	....	....	....	0.38	....	0.90	1.58	1.54
	....	0.53	1.46	0.69[b]	1.30	1.51	1.76	1.79	(0.86)
Casein	0.18	....	....	....	0.99	1.48	2.56	5.34	5.16
	....	1.77	13.34	8.05	10.54	11.44	13.18	16.70	5.13

[a] The figures for percentage loss in weight over P_2O_5 include the percentage loss in weight at 25 C. and 50 per cent relative humidity shown in column 5.

[b] Loss in 48 hr. at 25 C. and 50 per cent relative humidity.

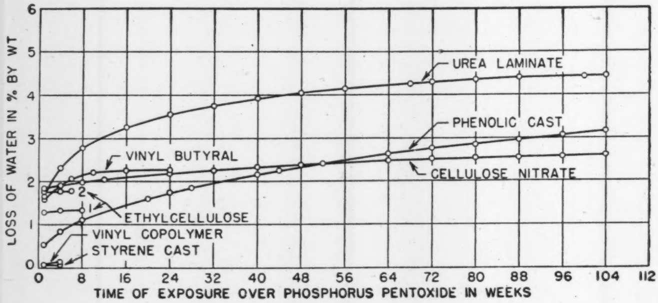

FIG. 2.—Desorption of Water by Plastics.

The curves in Fig. 1 reveal the marked differences in the amounts of moisture absorbed by the various plastics and the rates at which equilibrium conditions are approached by plastics immersed in water. The almost negligible amounts of moisture absorbed by styrene and vinyl chloride-acetate resins stand in sharp contrast to the avid affinity of the protein plastic for water. The curves show that some of the plastics had not yet reached equilibrium after 2-yr. immersion. The fourth specimen listed in Table VI for each plastic, that is, the specimen conditioned by exposure to 90 per cent relative humidity at 50 C. for 48 hr. followed by drying in an oven at 50 C. for 48 hr., was used for the long-time immersion tests. The net gain in weight for the 2-yr. period or the equilibrium state can be calculated from the data given in Table VI and Fig. 1.

DESORPTION OF WATER OVER LONG PERIODS

Data on the desorption of water by plastics upon long-continued exposure over phosphorus pentoxide are presented in Table VII and Fig. 2. The rates at which the equilibrium values are attained during exposure over phosphorus pentoxide and during immersion in water are quite different. Many of the plastics which had reached the equilibrium state in water or over phosphorus pentoxide prior to the 2-yr. limit were still changing in weight at the end of this period when exposed over phosphorus pentoxide or in water, respectively. The data in Table VIII on the changes in weight of the specimens during storage indicate that the continuous loss in weight of the cellulose nitrate and cast phenolic resin over phosphorus pentoxide, shown in Fig. 2, can be attributed in part to loss of volatile matter other than water.

TABLE VIII.—DIMENSIONAL CHANGES OF PLASTICS UNDER VARIOUS CONDITIONS AND WEIGHT CHANGES DURING STORAGE.

Material	Dimensional Change After Immersion in Water, per cent						Dimensional Change After Drying Over P_2O_5, per cent				Changes After Storage at 25 C. and 50 per cent Relative Humidity, per cent					
	Length[a] ±0.03			Thickness ±0.8			Length[a] ±0.03		Thickness ±0.8		Length[a] ±0.03		Thickness±0.8		Weight	
	48 hr.	8 weeks	2 yr.	48 hr.	8 weeks	2 yr.	8 weeks	2 yr.	8 weeks	2 yr.	48 hr.	2 yr.	48 hr.	2 yr.	8 weeks	2 yr.
Phenol-formaldehyde molded	0.05	0.65	1.35	0.8	3.2	3.2	......	......	......	......	......	......	...	......	......	......
Phenol-formaldehyde cast	0.07	0.36	2.08	0.3	1.6	3.9	−0.18	0.83	0.0	−0.8	......	−0.26	...	0.0	−0.14	−0.62
Phenol-formaldehyde laminated	0.00	0.16	0.13	0.5	2.2	9.0	−0.07	−0.03	0.0	0.0	0.03	0.07	0.0	0.8	0.25	0.65
Urea-formaldehyde molded	0.12	1.15	2.05	−0.8	4.8	2.3	......	......	......	......	......	......	...	......	......	......
Urea-formaldehyde laminated	0.03	0.26	0.30	0.8	3.1	3.0	......	−0.33	......	−1.5	0.00	0.00	0.8	1.6	0.09	0.13
Vinyl chloride-acetate resin	−0.02	0.13	0.03	−0.4	0.0	0.8	−0.07	......	0.0	......	−0.07	−0.07	0.0	0.0	0.01	0.00
Vinyl butyral resin	0.07	0.89	2.54	0.8	3.3	4.1	−0.36	......	−0.4	......	......	−0.03	...	0.8	0.13	0.19
Methyl methacrylate resin	0.12	0.40	0.30	0.0	0.7	0.0	......		......	......	0.03	0.03	0.0	0.0	0.04	0.02
Styrene resin molded	−0.13	−0.25	0.45	−0.8	0.0	0.0	......	......	......	......	......	......	...	......	......	......
Styrene resin cast	0.02	0.07	0.00	−0.4	0.0	−0.6	0.00	......	−0.3	......	0.00	0.00	0.0	0.0	0.00	0.00
Cellulose nitrate	0.35	0.30	0.07	0.6	0.8	0.8	−0.36	−0.80	−1.2	0.0	......	−0.56	...	−1.6	−0.36	−1.23
Cellulose acetate	0.71	0.40	0.53	0.8	2.3	1.5	−0.56	−2.09	0.0	−0.8	0.07	1.29	0.0	2.3	−0.22	......
Ethylcellulose No. 1	0.08	0.50	0.89	0.3	1.6	2.1	−0.10	......	0.0	......	0.00	0.00	0.0	0.0	0.14	0.27
Ethylcellulose No. 2	0.16	0.66	1.16	0.2	1.2	1.8	−0.15	......	0.0	......	−0.03	−0.07	0.0	−0.6	0.12	0.09
Cold-molded phenolic	−0.12	0.15	0.76	−0.3	0.0	0.8	......	......	......	......	......	......	...	......	......	......
Cold-molded bituminous	−0.08	0.35	1.21	−0.3	0.8	0.0	......	......	......	......	......	......	...	......	......	......
Casein	1.98	13.24	[b]	10.2	14.4	[b]	−1.58	−4.48	−2.2	−12.9	......	−1.54	...	4.9	0.26	0.74

[a] Diameter for molded specimens.
[b] Disintegrated after immersion for 44 weeks.

The curves in Fig. 2 are for the specimens which had been initially dried at 50 C. for 48 hr. and subsequently treated as shown in Table VII. The net losses in weight over phosphorus pentoxide can be calculated from the data given in Table VII and Fig. 2. The sum of the net loss when exposed over phosphorus pentoxide and net gain when immersed in water will indicate the total moisture-retaining capacity of any particular plastic under the conditions of these tests.

DIMENSIONAL CHANGES OVER LONG PERIODS

The expansions or contractions caused by changes in the moisture contents of the plastics are shown in Table VIII. The extent of the dimensional changes correlates well with the hygroscopicity of the plastic. Casein, which absorbs more moisture than any other plastic investigated, also shows the greatest changes in dimensions, and the hydrophobic materials, polystyrene and vinyl copolymers, show the least variation in dimensions under the different conditions.

Acknowledgment:

The authors express their appreciation to W. J. Clarke, C. A. Nash, and G. H. Mains, comembers of the Section on Water Absorption, which prepared the A.S.T.M. tentative method for measuring the absorption of water by plastics, for their interest and suggestions during the course of this investigation.

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MEASURING THE PLASTICITY OF MOLDING COMPOUNDS

By Robert Burns[1]

Synopsis

The need of a relatively simple device for measuring the plasticity of molding compounds has been recognized for some time. For those consumers of plastics who require some means of controlling the moldability of their compounds, a device is described which constitutes a simple and inexpensive supplement to the more precise methods. Data are given on a variety of compounds, both thermosetting and thermoplastic, and the effects on plasticity of such variables as moisture content of the powder, temperature, and pressure are discussed.

The use of capillaries and orifices for measuring plasticity of molding compounds dates back many years. The Bakelite Rossi-Peakes capillary device is familiar in the trade, and was used as an orifice apparatus at the Chicago plant of the Western Electric Co. at least fifteen years ago. Although circular capillaries are generally used, variously shaped orifices have been tried to simulate some particular phase of molding, such as the rectangular orifice for the study of gating in injection molding. It is obvious that such devices have contributed substantially to the progress of the plastics art.

Taking advantage of the knowledge gained through these pioneering efforts, the author has devised a plasticity die which is proposed as a relatively inexpensive supplement to existing methods. A description of the device with a discussion of the necessary operating technique and data illustrating its performance follow.

This apparatus, shown in Fig. 1, is essentially a $2\frac{1}{2}$-in. disk mold with capillaries of varying size extending downward from the disk. The capillaries are seven in number, the smallest, No. 7, being 0.015 in. in diameter and the largest, No. 1, 0.093 in. The split in the cone cuts each capillary exactly in half. The capillaries are 2 in. in length and are provided with an overflow cup for the measurement of very soft materials or for unusual molding technique involving high temperatures or pressures. The die gives a measure of plasticity which may be expressed numerically and produces a molded part for qualitative examination. It is not uncommon to find a material entirely satisfactory from a flow standpoint but unusable due to other defects as disclosed by a visual inspection of a molded specimen.

The testing technique for a thermosetting material is as follows:

The die, together with the plunger placed beside it, is heated between the press platens until the thermometer in the die shows a constant temperature of 300 F. The upper platen is raised,

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25 g. of molding powder is poured into the die, the plunger inserted and the press closed with a force of 4 tons ± 0.1 ton. This produces a pressure of approximately 1 ton per sq. in. The pressure should be applied promptly so that the figure of 4 tons is obtained within 20 sec. after filling the die. This pressure is maintained for 5 min. The specimen may be withdrawn hot. The end of each extruded rod of the specimen is tapped lightly with a pencil to remove any unmolded granules and the length of each rod is measured to the nearest 0.01 in. In order to express the plasticity of a thermosetting material as a simple arbitrary number for comparative purposes, the length of each rod is multiplied by the number of the capillary from which it was taken. The sum of the seven products is taken as the "plasticity index" of the material.

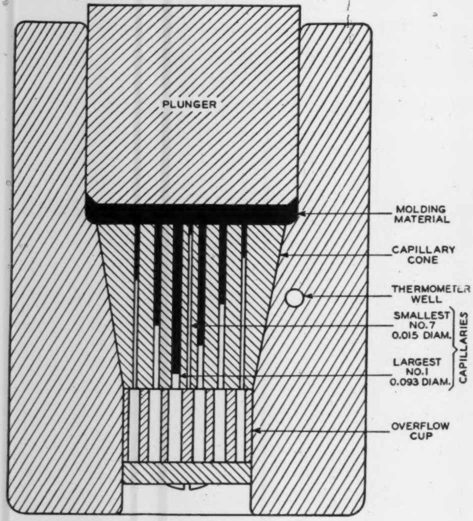

Fig. 1.

In the case of thermoplastic materials, the test procedure is the same with the following exceptions:

1. The temperature of the die must be adapted to the plastic properties of the material under test and must be less than that temperature which permits a complete filling of all the capillaries. The test is repeated three or more times at a series of temperatures below this level. The plasticity indices obtained as above described for thermosetting compounds, are plotted against the corresponding temperatures as in Fig. 4. The temperature on such a plot at which the plasticity index has a value of 20 is arbitrarily taken as the "plasticity temperature" of the material.

2. The die should be cooled to facilitate removal of specimen.

It will be seen that the plasticity of thermosetting materials as described above is expressed in entirely different terms from that of thermoplastic compounds. This is justified by the fact that in the thermosetting compounds the quality which one wishes to control in practice is a complex combination of its thermoplastic properties and the rate of its thermosetting reaction. By contrast, in the thermoplastic materials the purely plastic phenomenon is involved. In view of the fact that these tests are produced as empirical controls rather than to secure data for theoretical treatment, they must be judged by their practical utility rather than concordance to theoretical considerations of consistency. From a practical standpoint, the discrepancy is not serious. In the case of a thermoplastic compound whose general flow properties are known to the technician, a stipulated test temperature (as prescribed for the thermosetting materials) can be used. For example, in acceptance tests on successive batches of a given material it is entirely feasible to require that when tested at the stipulated temperature the plasticity index shall fall between prescribed limits.

The plastic art is still far from reaching a state at which a particular form of plasticity device can be adopted for universal use. The plasticity test herein discussed appears to possess a combination of features which should make it attractive to those who are concerned with the control of molding procedures. These features are:

1. The employment of seven capillaries of a range of diameters aids in predicting the flow of plastics in various parts of molds of complicated shape more accurately than is possible by flow through a single orifice.

2. Visual inspection of the molded part resulting from the test permits a qualitative observation of the inherent tendency of the material to produce a particular type of defect.

TABLE I.—REPRODUCIBILITY OF TEST RESULTS.

1. All day run on a wood flour filled phenolic—one batch—30 ton self-contained press—27 shots	Average 24.1	Max. 26.7	Min. 20.8
2. Same as above except press was hand pumped	24.3	28.1	19.0
3. Nine day-to-day tests on wood flour filled phenolic of different batches—30 ton self-contained press—each day's value an average of at least 5 shots	Average of Averages 24.6	Max. Avg. 25.7	Min. Avg. 23.5
4. Five day-to-day tests on wood flour filled phenolic of different batches—100-ton hand pumped press—each value an average of at least 5 shots	Average of Averages 25.2	Max. Avg. 27.7	Min. Avg. 22.9
5. Five consecutive day-to-day runs on cotton flock—wood flour filled phenolic—same batch—30 ton self-contained press—each value an average of at least 5 shots	Average of Averages 18.44	Max. Avg. 19.00	Min. Avg. 18.00
6. All-day run on cellulose acetate—100 ton hand press—9 shots	Average 27.4	Max. 31.9	Min. 23.3
7. All-day run on cellulose acetate—30 ton self-contained press—9 shots.	26.5	28.1	25.7
8. Same as above except press was hand pumped—9 shots	26.9	29.8	24.1
9. Four day-to-day tests on cellulose acetate—100 ton hand press—each value an average of at least 4 shots	Average of Averages 25.2	Max. Avg. 27.4	Min. Avg. 24.2

3. The scoring system proposed emphasizes the flow of material through narrow channels and consequently its adaptability to fill inaccessible parts of a commercial mold. This is accomplished by multiplying the length of the rod by the number of the capillary, the capillaries being numbered in inverse order of their diameters.

4. The device can be used for both thermosetting and thermoplastic materials, though the plastic values are expressed in different terms.

5. No observation of time is necessary in the testing of thermoplastics.

6. The plunger is the only moving part.

7. The die can be built for $300 or less.

The question which always arises in any discussion of a test method, especially where standardization between laboratories and between similar testing devices is concerned, is that of reproducibility. In this device, where there are no moving parts other than the plunger, the two variables (assuming identical compounds) are pressure and temperature. The degree of reproducibility therefore is almost entirely a function of how well these variables are controlled. This is shown by the difference in reproducibility where the measurements are made on a self-contained press and on a hand-operated press.

In the practical use of this die it has been found that the maintaining and measurement of the stipulated pressure constitutes the greatest difficulty in reproducing previous results on the same press, or in the interchangeable use of several presses, assuming, of course, that the capillaries are not in some abnormal condition at the steel-molding compound interface. It can be stated, however, that with any low-capacity press in good condition, and an accurate gage, the degree of reproducibility is adequate. Date illustrating the reproducibility of test results are given on Table I.

In any capillary or orifice device, the condition of the die at the steel-molding compound interface is always of importance. This also applies to commercial molds, and unusual conditions at the interface are occasionally designated in the molding trade by the term "mold poisoning." The term is generally used to describe any unique effect on the molding characteristics of a given compound by the interior surfaces of the mold. In changing from one phenolic, for example, to another with a different resin-filler ratio, the effect on the molding characteristics of the second compound is naturally slight. On the other hand, in changing from a phenolic to a cellulose acetate molding composition which involves an entirely different group of ingredients the effect may be marked. In plasticity work it is sometimes necessary to make several moldings of a new material before the influence of the material previously molded in the die is entirely erased. Experience with the multi-capillary die has shown that, just as in commercial molding, the die, if in continuous use, need not be cleaned or polished except on rare occasions. It should receive the same general care as any other mold, and for periods of idleness should be oiled in the usual manner to prevent rusting.

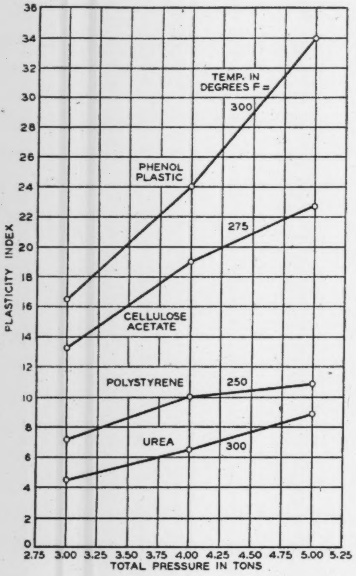

FIG. 2.

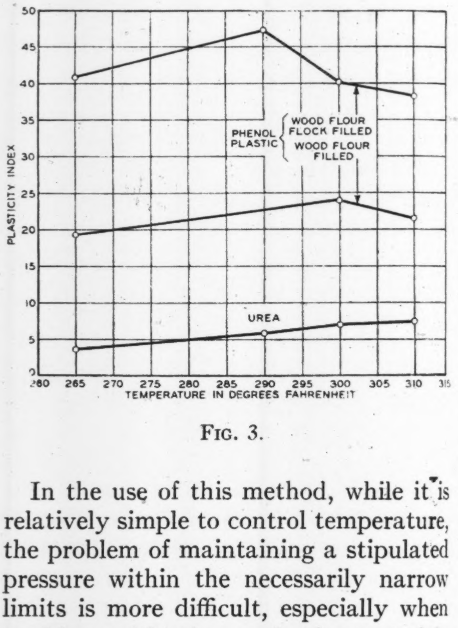

FIG. 3.

In the use of this method, while it is relatively simple to control temperature, the problem of maintaining a stipulated pressure within the necessarily narrow limits is more difficult, especially when working with thermoplastic materials for which close control of pressure should be maintained during the whole heating and cooling cycle. The use of high-capacity hand-operated presses with large ram diameters, in which small variations in line pressure result in large variations in total force, may yield unsatisfactory results. In Fig. 2 is shown the effect of pressure on the plasticity indices of a phenolic, two thermoplastic materials, and a urea compound. In Fig. 3 is given the effect of temperature on some thermosetting compounds, and in Fig. 4 its effect on typical thermoplastics.

It is well known that the plasticity of some of the molding compounds, by whatever method measured, is substantially affected by the moisture content of the powder. The effect of moisture on moldability is similarly well known to the molding trade. On Fig. 5 are given data illustrating this important effect. The materials covered are a high flock phenolic, a standard wood flour phenolic, a urea, and a cellulose acetate. Samples of polystyrene exposed to 90 per cent relative humidity for 14 days showed no change in plasticity because of the low moisture absorption of this material.

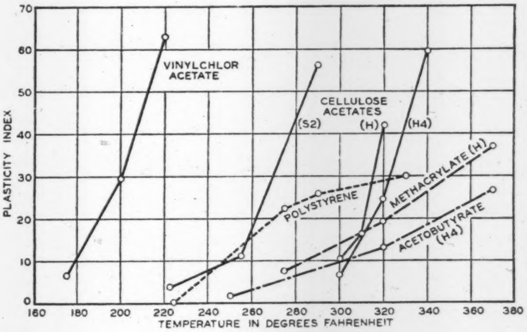

FIG. 4.

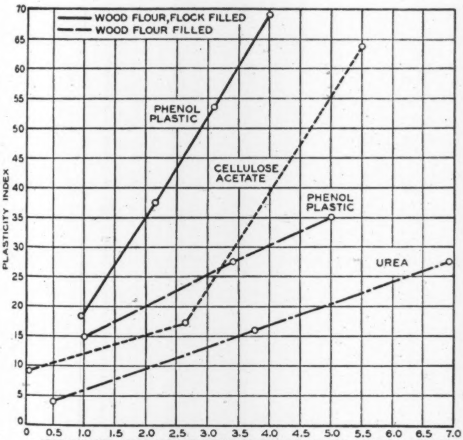

FIG. 5.

Since reproducibility depends, among other things, upon the diemakers' ability to duplicate the apparatus itself, it is of interest to compare capillary diameters in different dies. Table II gives the dimensions for three dies, two made by the same shop and the third by an entirely separate organization. Barring other factors such as mold poisoning or poor control of pressure or temperature, the spread of values of plasticity index obtained with the three dies should not be greater than the whole number 2.

In the use of this die a convenient

TABLE II.—COMPARISON OF CAPILLARY DIAMETERS IN THREE PLASTICITY MOLDS.
MOLDS 1 AND 2 MADE IN SAME SHOP.

Mold	Capillary Diameter, in.						
	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
No. 1	0.0920	0.0770	0.0631	0.0466	0.0320	0.0220	0.0156
No. 2	0.0940	0.0768	0.0613	0.0453	0.0300	0.0187	0.0147
No. 3	0.0925	0.0767	0.0620	0.0457	0.0301	0.0192	0.0146

method of determining empirical "flow times" for thermosetting compounds consists of mounting a dial gage between the upper and lower platens of the press. Measurement by stopwatch is made of the elapsed time from the instant the pressure gage registers 4 tons until the hand of the dial gage stops moving. For commercial phenolics this is usually in the neighborhood of 25 secs. The dial gage also furnishes interesting information on flow-velocity characteristics of thermoplastics.

A slide rule has been designed to expedite the calculation of plasticity index and minimize the possibility of error. It consists of 7 concentric disks, each one graduated in divisions corresponding to tenths of an inch filament length, making it possible to estimate hundredths. The calibration is such that the multiplying factor for each filament is implicit in the scale. In use, the zero line of the No. 2 disk (corresponding to the No. 2 filament) is set opposite the No. 1 reading, the zero of the No. 3 disk opposite the No. 2 reading, and so on, with a radial pointer set at the No. 7 reading on the inner disk and indicating simultaneously the Plasticity Index on the outer (No. 1) disk. It is illustrated in Fig. 6.

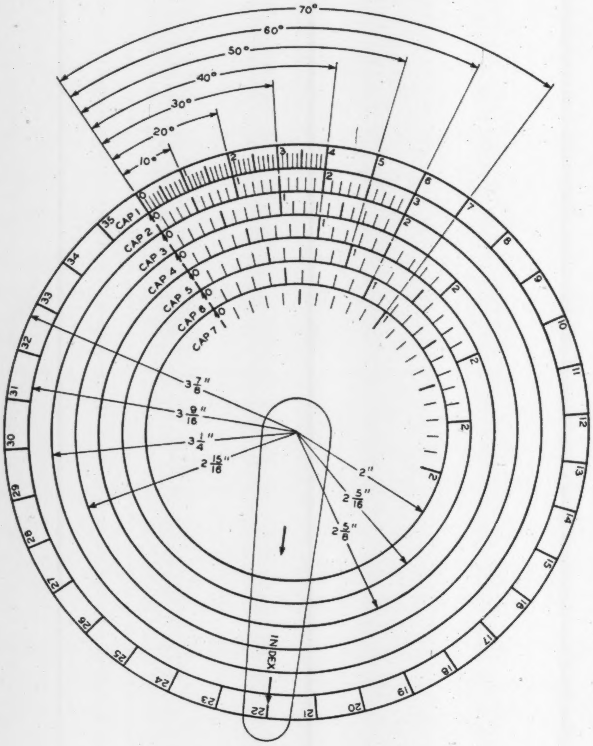
70°
60°
50°
40°
30°
20°
10°
CAP 1
CAP 2
CAP 3
CAP 4
CAP 5
CAP 6
CAP 7
3 7/8"
3 9/16"
3 1/4"
2 15/16"
2"
2 5/16"
2 5/8"
INDEX
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FIG. 6.

THE REDUCTION OF SECONDARY RADIATION AND OF EXCESSIVE RADIOGRAPHIC CONTRAST BY FILTRATION[1]

By Herman E. Seemann[2]

Synopsis

To secure the best radiograph, it is often necessary to mask irregular objects, or to make separate exposures for the thick and thin parts. The need for such procedures can be lessened, or avoided entirely, by the use of a filter in the X-ray beam to diminish subject contrast and to decrease the amount of secondary radiation reaching the image from portions of the cassette not covered by the object. Berthold has shown that when a lead filter is used between the object and the film, an additional filter of tin removes most of the undesirable K radiation originating in the lead. The present experiments show that still further improvement in radiographic quality can be made by placing the filter as near the X-ray tube as possible. With the filter in this position, the influence of its secondary radiation is negligible so that no additional filter to absorb its secondary radiation is required. Radiographic sensitivity is diminished somewhat by the filtration technique, but this may not be a serious handicap in many types of work. Minor advantages of the "distant-filter" technique are: The specimen-film distance is less than when the filter is on the cassette; possible defects in the filter are blurred out so that they will not be confused with voids in the specimen; and small filters placed on the tube shield are more convenient to handle. Exposure data are listed for several representative subjects. A variable-intensity illuminator is advantageous for the examination of the wide range of densities often occurring in radiographs of irregular objects.

The problem of making a satisfactory radiograph of small or irregular objects is resolved into preventing overexposure in thin or open regions and, at the same time, obtaining a satisfactory record of thick parts. If economic considerations can be ignored, two general methods are available which yield at least as good radiographs as can be obtained by modern methods for the same thickness of the material in the form of large plates. A small object which does not have much thickness variation in the direction of the rays may be masked by sheet lead, barium clay, or other suitable absorber. Obviously, some time will be involved in making the necessary preparations, but excellent radiographs can be produced if reasonable attention is paid to the other factors, such as distance and kilovoltage. A specimen with large thickness differences, that is, one which has high subject contrast, may be radiographed by exposing thick parts while thinner parts are blocked off, and then uncovering the latter for other suitable exposures. Instead of combining all these in one radiograph, separate radiographs may be made for each thickness. These methods cannot be criticized on technical grounds, but in almost all industrial work economy is one of the most important considerations. For this reason, techniques have been developed which are more economical.

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[1] *Communication No. 758* from the Kodak Research Laboratories.

[2] Physicist, Physics Dept., Research Laboratories, Eastman Kodak Co., Rochester, N. Y.

Carbon tetrachloride,[3] water solutions of lead salts, and other liquids[4] have been used as masking and contrast-reducing agents with some success. Their use is obviously restricted. Fine metallic shot[5] has also been used to fill in cavities or to compensate for the disturbing irregularities in the bead of a weld. This material is available in a fine enough grade so that individual particles do not show in the radiograph. It is usually more convenient to use than the liquids.

The use of a uniform metal sheet in the X-ray beam to reduce subject contrast[3] is well known. The principle of this technique is that the X-ray intensity is reduced by a much greater fraction through the thin portions of the specimen than it is through the thick parts. The filter removes some of the softer components which penetrate thin sections, but, since the radiation is already hardened considerably in passing through thick regions of the specimen, the filter has less effect. Consider, for example, a piece of steel with a minimum thickness of 3 mm. and a maximum thickness of 22 mm. A satisfactory radiograph may be made with the addition of 6 mm. of steel as a filter. The use of this filter will necessitate a somewhat longer or higher voltage exposure, but all parts may be rendered in densities which are within the range of an adjustable, high-intensity illuminator.[6] In reducing contrast, the filter will, of course, diminish the radiographic sensitivity, but this limitation may well be offset by the convenience and rapidity of the technique. The use of a tapered wedge in the radiography of fillet welds[7] is a variation of the filter method for reduction of subject contrast. If a "perfect" radiographic examination is essential, separate exposures should be made for each thickness, heavy filtration being definitely undesirable. Selection of a technique will depend therefore upon a consideration of all factors involved because the most sensitive method is likely to be the most expensive. The filtration technique will almost certainly involve some sacrifice of sensitivity in the interests of economy, but the results may be adequate for the purpose.

Assuming that this technique is to be applied, it is reasonable to inquire whether there is any choice in the *method* of application which will minimize its limitations. Berthold[8] has used sheet lead as a filter between the specimen and the cassette in the radiography of thick sections. Recognizing the fairly penetrating nature of the K radiation from lead, he introduced an additional filter of tin between the lead and the cassette to absorb this radiation. His experimental results indicated a definite improvement resulting from the use of the tin filter. Since the Compton effect accounts for considerable secondary radiation under the usual conditions of industrial radiography, a filter absorbing the *characteristic* radiation of lead still transmits much of the *total* secondary radiation. In the course of the present investigation, trials of Berthold's tin and lead combination showed a definite improvement over the same filtration with filter positions reversed, that is, with the lead nearer the cassette. All matter emits both Compton and characteristic secondary rays when irradiated with sufficiently hard X-rays, so that, while Berthold's argument is sound, the Compton scattering from both the tin and the lead still presents a problem.

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[3] W. L. Fink and R. S. Archer, "Radiography as a Tool in the Metal Industry," *Transactions*, Am. Soc. Steel Treating, Vol. 16, p. 551 (1929).

[4] A. St. John and H. R. Isenburger, "Industrial Radiography," p. 151, John Wiley and Sons, New York, N.Y. (1934).

[5] C. D. Moriarty, "The Use of Metallic Shot in X-raying Steel," *General Electric Review*, Vol. 42, No. 3, March, 1939, p. 109.

[6] R. G. Tobey, "Recent Progress in Industrial Radiography," *The Iron Age*, Vol. 145, February 29, 1940, p. 27; March 7, 1940, p. 64.

[7] J. C. Hodge, "Radiography in the Welding Shop," Symposium on Radiography and X-ray Diffraction, p. 53, Am. Soc. Testing Mats. (1936). (Symposium available as a separate publication.)

[8] R. Berthold, "Improving Radiographic Technique by Filters," *Archiv* für das Eisenhüttenwesen, Vol. 8, No. 1, p. 21 (1934).

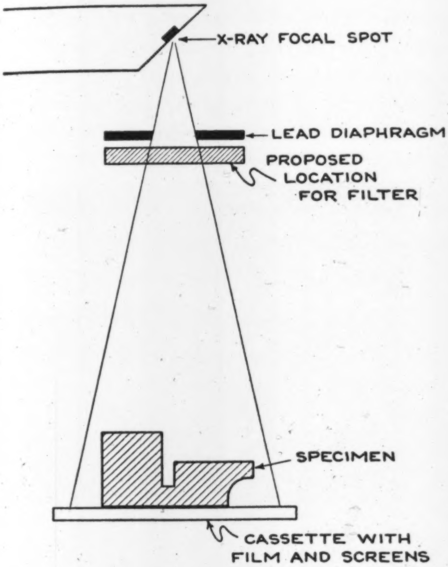

FIG. 1.—Schematic Diagram for the Making of a Radiograph of an Irregular Specimen by the "Distant-Filter" Technique.

In developing a geometrical method for measuring secondary radiation[9] under idealized radiographic conditions, it was found experimentally that the secondary radiation from a metallic plate specimen near the X-ray tube had only a small fraction of its intensity with the plate near the film, both measurements being made at the film in its fixed position.

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[9] H. E. Seemann, "Secondary Radiation Intensity as a Function of Certain Geometrical Variables," *American Journal of Roentgenology*, Vol. 39, No. 4, April, 1938, p. 628.

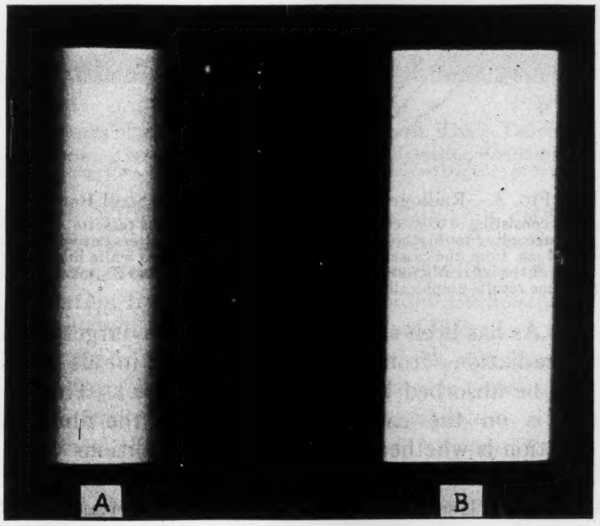


FIG. 2.—"Silhouette Radiographs" of a Lead Bar 0.9 cm. Thick and 4 cm. Wide Taken at 200 kv.p. Using a Copper Filter 1.0 cm. Thick.

In a radiograph *A*, the copper filter was on the cassette, and in radiograph *B*, it was 67 cm. from the cassette.

This led to the belief that an improved radiograph might result when using the filtration technique if the filter were placed as near the tube as possible, it being assumed that the X-ray beam is diaphragmed to the smallest practicable size (see Fig. 1). Fink and Archer[3] have mentioned the use of the filter in different positions but they did not make comparative tests. As has been stated,[4] some secondary radiation from the specimen itself will be absorbed by the filter if the filter is on the cassette. The important question is whether more radiation will be *contributed by the filter* than is absorbed from the specimen. This must be answered by experimental comparisons.

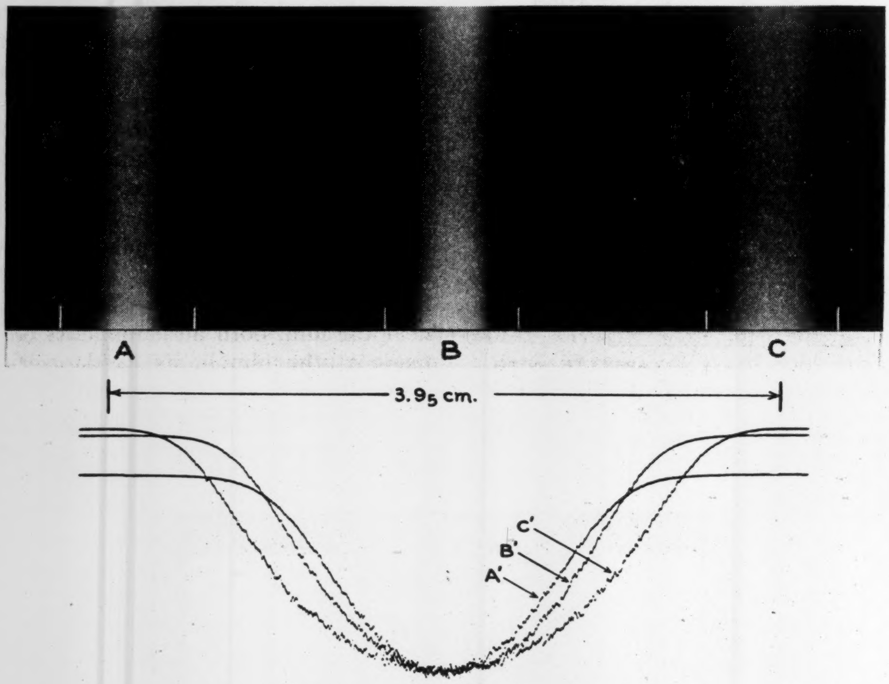
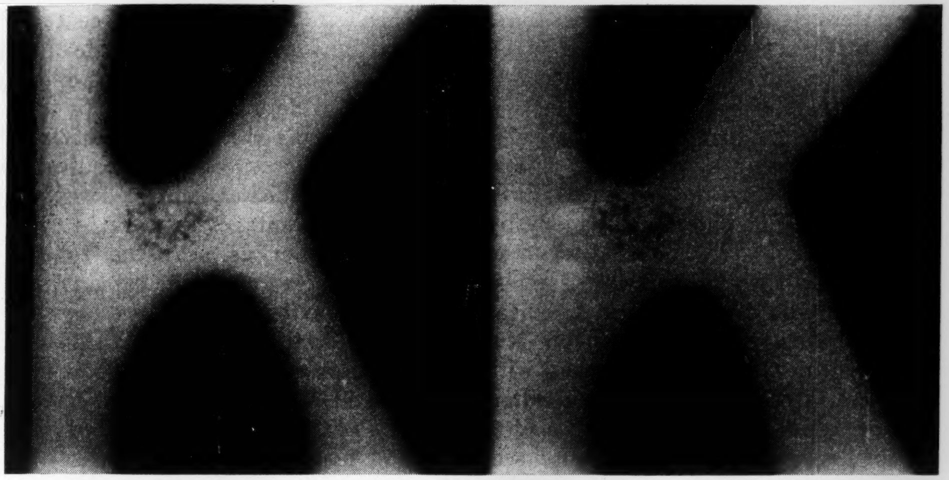


FIG. 3.—Radiographs of a 3.8-cm. Square Steel Bar.

A was taken with a filter consisting of 0.088-cm. tin plus 0.078-cm. lead on the cassette, the lead being in contact with the cassette. *B* was taken according to Berthold's technique with the same filters but with the tin in contact with the cassette. The filters were 67 cm. from the cassette in radiograph *C*. The short white lines on the radiographs indicate the true limits of the image of the bar. Microdensitometer records, *A'*, *B'*, and *C'*, taken across the respective radiographic images, show the same results graphically.

Figure 2 shows a "silhouette radiograph" of a lead bar 0.9 cm. thick and 4 cm. wide taken at 200 kv.p. using a copper filter 1.0 cm. thick. This is an extreme case cited for illustrative purposes. The lead bar was not appreciably penetrated. In radiograph *A*, the copper filter was on the cassette, and in radiograph *B* it was 67 cm. from the cassette. The target-film distance was 1 m. (This target-film distance is the value used in all of the experiments reported here.) The lead bar was about 4 cm. from the film in both cases. In fact, all conditions were identical except the position of the filter. It is obvious that the undercutting of the specimen by the secondary radiation from the copper filter may be serious when the filter is on the cassette.



A B

FIG. 4.—Radiographs of a Test Casting of Iron 2.9 cm. Thick Taken at 200 kv. p.

Width of arms of K about 4 cm. *A* was taken with 0.088-cm. tin on the cassette, *B* with the same filter near the tube Note that, because of undercutting, the shrinkage porosity *appears* to be nearer the edge of the casting in *A* than in *B*.

In order to radiograph thick sections of steel or brass, considerable filtration is necessary to prevent excessive undercutting of secondary rays around the boundaries of the thick portions of the specimen. As a matter of convenience, thin lead may be used instead of the more bulky quantity of copper which would be necessary for the same absorption. In Fig. 3, radiograph *A* was taken with a combined lead and tin filter on the cassette but with the *lead nearer the cassette* so as to have a radiograph for comparison with one taken according to Berthold's technique which calls for placing the *tin nearer the cassette* to absorb the K radiation from the lead filter. The tin used was 0.088 cm. thick which absorbs about 80 per cent of the shortest of the lead K radiations. It therefore absorbs more than this proportion of the total K spectrum. Radiograph *B* was taken according to Berthold's method and shows a definite improvement over *A*. Radiograph *C* was taken with the same filters 67 cm. from the film (33 cm. from the focal spot). This radiograph shows an improvement over both of the preceding. Secondary radiation originating in the filter is reduced to a small fraction of its value in the former cases because of the great *distance between secondary source and film*. Thus, a change in geometrical relationships is responsible for the radiographic improvement, and the *quality* of the secondary radiation from the filter is unimportant. Microdensitometer records A', B', and C' were taken of the respective radiographs and verify the conclusion that the "distant-filter" method is an improvement over either of the other two.

It might be assumed that consideration should be given to the selection of a filter which, for the necessary absorption, would yield a minimum of secondary radiation. Accordingly, thicknesses of copper, tin, and lead having equal absorption were placed side by side between the cassette and a 0.9-cm. lead bar. These filters were all clamped against the lead so as to equalize geometrical factors. An exposure was made at 150 kv. p. The minimum amount of secondary effect was observed for the tin, with that for copper and lead about equal. Equality for copper and lead is attributed to possible compensation for a deficiency of Compton radiation in the case of lead by its intense K radiation. Presumably, tin has a low total output of secondary radiation because it has too high an atomic number to emit strong Compton radiation and too low an atomic number to emit strong, penetrating K radiation. Relationships may be quite different at other kilovoltages. The noteworthy point is that when these filter materials were placed near the X-ray tube, the secondary intensity at the film was so low that a distinction could not be made radiographically between such small quantities. The conclusion is that the choice of filter materials may be determined chiefly by convenience.

Figure 4 shows radiographs of a test casting of iron 2.9 cm. thick, taken at 200 kv. p., 10 ma. minutes, on No-Screen Film with lead screens, with 0.088-cm. thickness of tin as a filter. Radiograph *A* was taken with the filter on the cassette and shows more secondary radiation extending under the specimen than radiograph *B* which was taken with the filter 67 cm. from the film.

The chief advantage of the distant-filter technique is that by substantial reduction of undercutting of the specimen by secondary radiation from the filter, the radiograph has a diagnostic value over a greater area of the image. Within the limits of the experiments tried for this study, no gain in radiographic sensitivity has been observed in regions which did not show undercutting. This undercutting by secondary radiation reduces contrast wherever it occurs and therefore interferes with satisfactory rendition of detail. In the central portions of the image, under full specimen thickness, the removal of secondary radiation by a filter on the cassette seems to be largely offset by its own contribution in so far as radiographic appearances can be used as a criterion. Of course, *any filter* at *any location* in the X-ray beam will reduce sensitivity to some extent because its presence is the equivalent of additional specimen thickness, and sensitivity is expressed in terms of *specimen* thickness, *not total* thickness. In consideration of this principle, it has been gratifying to find no difficulty in detecting 2 per cent additional thickness on the tube side of the specimen and, in many cases, 1 per cent was barely detectable. This is consistent with the fact that *approximately* one-half of a specimen's equivalent thickness is added as a filter.

Minor advantages associated with using the filter as near the tube as possible, instead of on the cassette, are: (1) A reduction in the *specimen-film distance;* (2) the *blurring-out* of possible damaged or defective areas in the filter itself; and (3) the convenience in handling *filters of small area* placed in a suitable holder on the tube shield. The limited number of observations made in this investigation does not preclude the possibility that for some particular case a filter on the cassette might be better than one near the tube, but it is felt that this is more likely to be the exception than the rule. Little practical difference in the two methods is observed for cases requiring as light a filter as 1 mm. of copper, for example, but the minor advantages above mentioned indicate that the distant-filter technique would be preferred.

The conditions for the radiography of small, irregular objects are so varied that it is impossible to give a complete exposure table. Furthermore, a certain amount of variation in opinion is to be

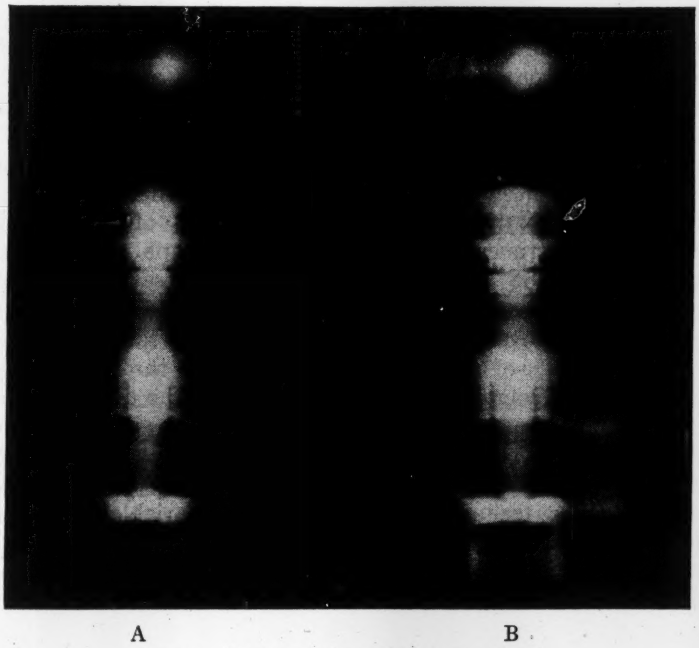


FIG. 5.—Radiographs of ¼-in. Brass Valve.

A with 0.111-cm. lead and 0.088-cm. tin on the cassette, the latter filter being adjacent to the cassette. *B* was taken with the same filters 33 cm. from the tube and the specimen separated from the cassette a distance equal to the filter thickness so that *definition* would be the same in both cases.

TABLE I.

Article	Material	Maximum Thickness, cm.	Filter Thickness, mm.	kv. p.	Milliampere Minutes	Film	Screens	Remarks
Cast bar, square section	Aluminum alloy	3.2	1.5 copper	110	10	Eastman No-Screen	Lead alloy foil	Alloy screen, 6 per cent antimony, 94 per cent lead. Front screen 0.013 cm. thick.
Casting, cup shape	Aluminum alloy	5.4	3.0 copper	130	10	Eastman No-Screen	Lead alloy foil	
Round rolled bar	17S-T Aluminum alloy	3.8	1.5 copper	110	10			
Round rolled bar	17S-T Aluminum alloy	6.7	3.0 copper	140	10			
Eastman interval timer	Brass and steel		2.0 copper	130	4			
Bourdon gage	Brass	1.4	6.0 copper	180	4			
¼-in. valve	Brass	2.5	10.0 copper	200	20			
K casting	Iron	2.9	5.0 copper	200	10			
Round rolled bar	Steel	1.9	3.0 copper	170	10			
High pressure valve	Steel	2.5	5.0 copper	200	10			
Flat bar	Lead	0.9	10.0 copper	200	20	Eastman No-Screen	Lead alloy foil	"Silhouette," bar not penetrated.
Square rolled bar	Steel	2.5	7.0 copper	150	10	Eastman Ultra Speed	Eastman Industrial	Calcium tungstate
Square rolled bar	Steel	3.8	0.78 lead	180	10	Eastman Ultra Speed	Eastman Industrial	
Square rolled bar	Steel	5.1	2.0 lead	220	40			

Target-film distance was 1 m. and filter-film distance was 67 cm. for all exposures. Projected focal spot size was about 1 cm. square.

expected in judging the quality of radiographs of complicated forms. Table I gives techniques which produced satisfactory radiographs for several objects. It is a rough guide but may serve a useful purpose as such. Most of the specimens chosen were of simple form, for example, square bars, to facilitate judging the original experimental radiographs and to make the data more readily interpretable by others.

Many light specimens involving thicknesses of only a centimeter of aluminum, for example, may be radiographed with no other filter than the front lead screen. Probably anything which can be radiographed below about 90 kv. will not require a special filter technique.

Different thicknesses of metal in the same object may yield a large range of photographic densities in one radiograph. Definite limits cannot be assigned to these densities because of the various characteristics of different X-ray films, but the most useful range extends from approximately 0.8 to 2.5 or 3.0. Variable illumination should therefore be available in order to obtain the maximum amount of information from a given radiograph. For example, if the illumination is satisfactory for a density of 1.0, it must be about thirty-two times greater for the same amount of light to reach the eye through a density of 2.5. Fortunately, the eye has a fair range of accommodation so that the variation available in the illuminator does not need to correspond exactly to the densities. Variable illumination with suitable masks will prove especially useful in examining radiographs of miscellaneous subjects.[6]

DISCUSSION

Mr. Herbert R. Isenburger[1] *(presented in written form)*.—Mr. Seemann's paper indicates the vast amount of work that has been done to improve a bothersome condition, and yet there remains a tremendous amount of systematic research still to be done before we can be entirely satisfied with our results.

While Mr. Seemann confines his paper to secondary radiation, it is not always easy to distinguish between secondary and scattered radiation. It is thus generally understood that the Compton effect which Mr. Seemann mentions frequently is caused by scattered radiation. It is particularly noticeable with high voltages between 250 and 650 kv. Unfortunately, the highest voltage mentioned in the paper was 220 kv. which seems to indicate that the most important range of scattered radiation has not been dealt with in the present investigation. It would be most useful to have this subject discussed in a subsequent paper.

Restricting our comments to secondary X-radiation, it may be mentioned that modern X-ray tubes incorporate electron filters,[2] and metal discharge chambers built around the target,[3] as well as automatically interchangeable filters[4] built into the protective tube shield not only to eliminate all undesirable forms of radiation but at the same time to utilize some of this waste radiation to improve the quality of the effective rays. This is done to such an extent that 200-kv. radiation corresponds to 300 kv. from ordinary Coolidge tubes.[5] With this modern tool, filters in front of the film holder are not necessary unless (1) we are dealing with irregularly shaped objects, (2) we wish to eliminate K_α lead radiation from a solution, or (3) we are using X-rays generated above 250 kv.

The last paragraph in Mr. Seemann's paper mentions film densities ranging from 0.8 to 2.5 or 3.0 as the most useful portion of the gradation curve. Granted that variable illumination improves and facilitates the reading of negatives—this, however, can only be considered as an emergency measure. It is universally acknowledged that the best range lies between 0.4 and 1.25 with 0.75 to be aimed at in exposure technique and 1.8 as the upper limit where sufficient details can still be detected with the aid of forced illumination. In any case, the limit of a gradation curve is reached at about 2.65, which is practically black, and it never reaches 3.0.[6] The accompanying Fig. 1 shows an ordinary density curve. Photographic density is defined as the logarithm to the base 10 of the ratio of the light incident on a photographic deposit to the light transmitted by that deposit. It is usually measured by a Martens photometer. A measured exograph of an aluminum wedge specimen is reproduced in the accompanying Fig. 2.

Naturally, it is more economical to

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[1] President, St. John X-ray Service, Inc., Long Island City, N. Y.

[2] A. Bouwers, "Economy and Quality in Radiology," *British Journal of Radiology*, Vol. 5, p. 311 (1932).

[3] J. H. Van Der Tunk, "Secondary Electrons in X-ray Tubes," *Physica*, Vol. 12, p. 274 (1932).

[4] G. E. Bell, "Effect of Filtering on Spectrum of an X-ray Beam," *Journal*, Inst. Production Engrs. (British), Vol. 19, No. 2, p. 42 (1940).

[5] H. R. Isenburger, "X-ray Tubes for Industry," *Electronics*, Vol. 11, p. 20 (1938).

[6] O. J. Baltzer and J. E. Nefe, "Photographic Reversal with X-rays," *Physical Review*, Vol. 57, p. 1048 (1940).

work with exographs of lower density. At the beginning of his paper, Mr. Seemann rightfully mentioned the fact that economy is one of the most important considerations in almost all industrial work. From this point of view, may we suggest that a paper be prepared on the *beneficial* results which can be obtained by secondary and scattered radiation. I am thinking particularly of certain results obtainable with and without lead backing of the film, having the film close up to the object and leaving a little space between object and film, and similar considerations which may not necessarily be best radiographic technique but which will be sound economy without appreciably impairing the quality of the results.

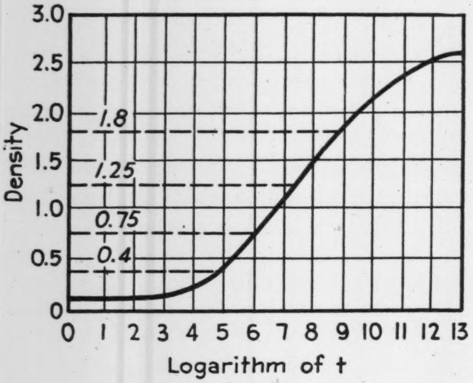

FIG. 1.—Density Curve (Eggert).

John Eggert, "Roentgenphotographie," S. Hirzel Verlag, Leipzig (1935).

MR. H. H. LESTER.[7]—I should like to remark that the under-cutting that Mr. Seemann has been talking about has bothered almost every one who had to radiograph small objects. There are various methods of overcoming it, I believe Mr. Isenburger at one time pointed out that immersing in liquid of suitable optical density gets away from it satisfactorily, but this method is not always convenient. We have been putting the filter over the top of the object and have found considerable improvement. Mr. Seemann's procedure seems to be better since his results are superior to any we have obtained.

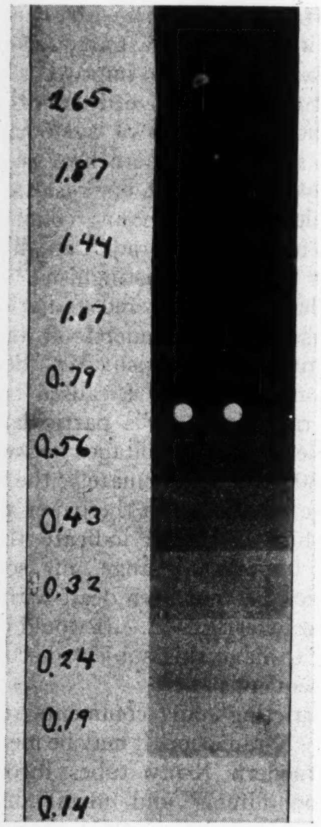

FIG. 2.—Measured Exograph of an Aluminum Wedge Specimen.

Courtesy V. B. Sease, Director of the Redpath Laboratory, Du Pont Film Manufacturing Corp., Parlin, N. J.

MR. L. W. BALL.[8]—I am impressed by the directness of the tests that Mr. Seemann has applied. The same idea should be extended to the radiation passing through the film and to radiography at higher voltages. In Mr. Seemann's experiments the radiation scattered between the tube and the film was studied. The radiation which passes right through the cassette must strike near or distant material and some radiation is scattered back to the film. Mr. Seemann's results suggest that the common use of thick lead a few inches behind the film is the worst possible arrangement. To test this, a rough experiment was made in our laboratory in which the 600-kv. rays were stopped by three materials: lead, lead covered with tin, and a lead-tin-copper-aluminum assembly. The results were that putting the tin there reduced the amount of back scattered radiation, but the further addition of lighter elements produced no effective change.

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[7] Senior Physicist, Watertown Arsenal, Watertown, Mass.

[8] Division of Physics and Electrical Engineering, National Research Council, Ottawa, Ont., Canada.

One other appropriate consideration is X-ray protection. The amount of secondary radiation inside a room in which an X-ray tube is operating depends on the material with which the room is lined. Many rooms are lead lined. It appears that this is not the best condition and an improvement would occur if the room were lined with tin; possibly covering the lead with plaster is a satisfactory solution.

MR. HERMAN E. SEEMANN.[9]—Mr. Ball is undoubtedly correct in suggesting that there would be less secondary radiation within an X-ray room if the lead lining were covered with tin. Is it not true that in industrial radiography, at least, the protection is for the benefit of those outside the room?

MR. BALL.—Reduction of secondary radiation inside the enclosure is required, either to protect the patient in X-ray therapy or to improve the film in industrial practice.

MR. SEEMANN.—That is true, but I think that the choice of a suitable technique makes the type of lining a minor consideration. Proper diaphragming of the primary beam and providing a fair distance from the subject to the floor or wall should make secondary effects negligibly small. It is particularly important that no appreciable part of the original primary beam goes past the subject to strike the wall. This would of course, excite much more secondary intensity than the beam which passes *through* the specimen because of its greater intensity.

Cassen and Corrigan have described[10] a cellular grid so constructed that it prevents back-scattering of most of the incident radiation. This grid has a more or less honeycomb structure, the sides and bottoms of the cells being of lead. It thus acts as a kind of radiation trap. It could be placed in any location where an X-ray beam normally impinges.

I regret that I did not make any measurements of back-scatter in connection with the work reported today. Extrapolating from some points which I did test, I agree with Mr. Ball. I suppose the lead inside the cover of most cassettes contributes a little to the useful image if it is near the film. The closer the lead and film, the better will primary and secondary images coincide in sharpness of outline.

Referring to Mr. Isenburger's comments, I believe there is a misunderstanding in terminology but I feel that I am in agreement with the accepted nomenclature of physics when I use the term "secondary radiation" for *any radiation* excited in *any manner* by a certain primary beam.[11] Compton scattering gives rise to one kind of secondary radiation; fluorescent X-radiation is another kind. In a problem like the one under discussion, it seems better to use the general term since no direct tests were made to classify the radiations.

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[9] Physicist, Physics Dept., Research Laboratories, Eastman Kodak Co., Rochester, N. Y.

[10] B. Cassen and K. E. Corrigan, "The Reduction of Back-Scatter from Roentgen Beams," *American Journal of Roentgenology*, Vol. 40, p. 608 (1938).

[11] Compton and Allison, "X-rays in Theory and Experiment," p. 12, D. Van Nostrand Co., New York, N. Y. (1935).

Mr. Isenburger points out that when a certain type of X-ray tube is used, filters are not required "unless (1) we are dealing with irregularly shaped objects . . ." The technique I have described offers its greatest usefulness in the radiography of irregular forms; hence, filters are required.

Experience and the form of the characteristic curve of modern X-ray films indicate that a total H and D density of 0.4 is not high enough for maximum radiographic sensitivity. (This does not mean that such a density is of no use!) Parts of an image having a density of 0.4 will have a lower contrast than regions of somewhat higher density. Under *average* illumination, it is difficult to see much detail in densities above 1.8, but this limitation is not due to the characteristic of the film. High intensity illumination is easily provided for higher densities. With sufficient exposure, densities well above 3.0 are attained, though no one would contend that they are radiographically useful. Some types of film other than X-ray film may not yield this density. The curve which Mr. Isenburger has selected from Eggert's book[12] does not represent present-day X-ray films. It may be well in this connection to consider Fig. 22, p. 36 and Fig. 29, p. 44 in Eggert's later book.[13]

I am not sure just what beneficial results may be derived from secondary radiation unless we admit the special case of utilizing the emission from lead-foil screens. This is, of course, a very useful application but also a very special one. It seems to me that, in general, secondary radiation is something to be disposed of in the most efficient manner.

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[12] John Eggert, "Roentgenphotographie," S. Hirzel Verlag, Leipzig (1935).

[13] John Eggert, "Einführung in die Roentgenphotographie," S. Hirzel Verlag, Leipzig (1936).

PRECISION RADIOGRAPHY—II

By Gilbert E. Doan[1] and Winston H. Sharp[2]

The minimum crack width which could be detected in steel specimens from $\frac{1}{4}$ in. thickness up to 4 in. thickness was reported last year in the paper presented before the Society by Vordahl and one of the present authors.[3] In those experiments only one crack depth was used, namely $\frac{1}{4}$ in., except in one or two cases where $\frac{1}{2}$ in. depth was used. In the present study, crack depth was varied as well as crack width and specimen thickness. The limits are plotted in the graphs. These results show that there are definite limits beyond which internal flaws will not be revealed by these radiographic methods. They substantiate further the observation made last year, namely, that depth sensitivity limits, such as those determined by Norton and Ziegler, are not absolute but must be expressed also in terms of crack width.

Experimental Methods

The radiant source used was a capsule 3 mm. in inside diameter, containing 25 mg. of radium sulfate placed at our disposal by the Radium Chemical Co. of New York. About this source were arranged the test pieces at the correct focal distances (Fig. 1). They were oriented with the crack pointed directly at the source but the crack was at the surface of the specimen farthest from the film. The cracks were formed as spacings between the carefully machined edges of abutting plates. The focal distances were based upon the work of Doan and Vordahl[3] and corrected for the difference in source size by means of the geometrical relationship proposed by Doan and Young.[4]

The films were overexposed 50 per cent, developed in potassium iodide developer and were viewed in both sunlight and in a standard viewing cabinet. When a negative contained little or no shadow, duplicate films were viewed superimposed.

Results

In Fig. 2 are three graphs which give the results of the 36 radiographs used. In each of the three graphs the crack depth is maintained constant, namely, $\frac{1}{8}$ in., $\frac{1}{4}$ in., and $\frac{1}{2}$ in. The crack width is plotted as the ordinate and the total steel depth as the abscissa. Since the total steel depth is one of the variables, the focal distance is also variable from 4 in. (source to flaw) for $\frac{1}{2}$ in. of steel to 16 in. distance for 4 in. of steel. A crack width of 0 in. means that the accurately machined faces forming the crack were pressed together tightly.

For example, for $\frac{1}{8}$-in. cracks, it is shown that a crack 0.003 in. wide in 2 in. of steel will be detected. If the crack width is reduced to 0.002 in., the crack will not be detected under these conditions. For $\frac{1}{4}$-in. crack depth, a crack

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[1] Professor of Metallurgy, Lehigh University, Bethlehem, Pa.

[2] Assistant Metallurgist, Research Dept., Vanadium Corporation of America, Bridgeville, Pa.

[3] Gilbert E. Doan and Milton B. Vordahl, "Fine Focus Radiography—Gamma Rays," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 741 (1939).

[4] Gilbert E. Doan and Shang-Shoa Young, "Gamma-Ray Radiography," *Proceedings*, Vol. 38, Part II, p. 292 (1938).

0.002 in. wide is readily detected in 2 in. of steel, whereas with cracks ½ in. deep a 0.002-in. wide crack is readily detected in 3 in. of steel thickness.

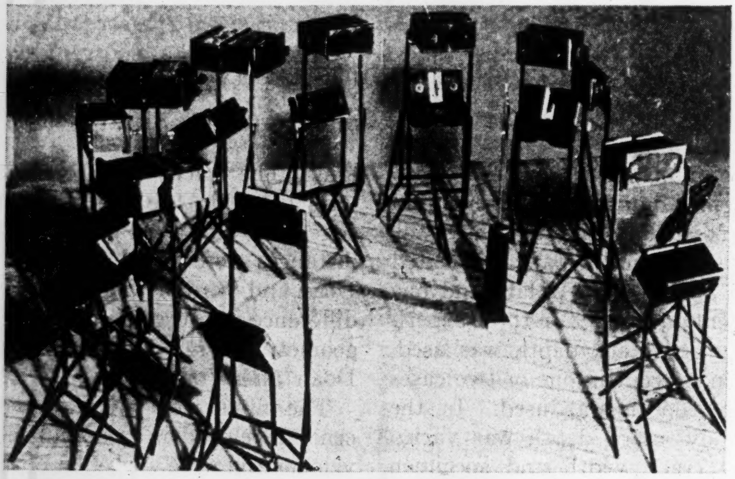

FIG. 1.—Arrangement of Specimens on Supports.

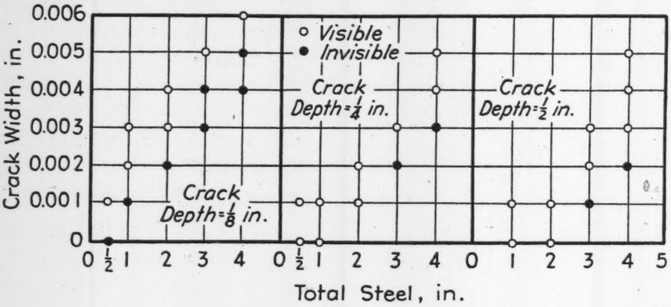

FIG. 2.—Results of 36 Radiographs.

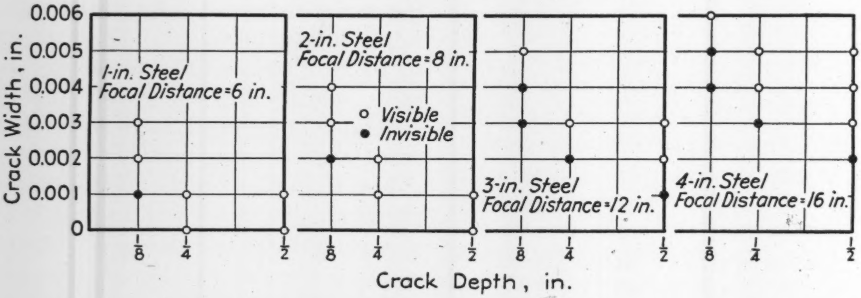

FIG. 3.—Relation of Crack Width to Crack Depth.

In Fig. 3 we have crack width plotted against crack depth, with the total depth of steel (and consequently the focal distance) held constant. Reference to any of these graphs will reveal that a crack of given width may go undetected when it is but $\frac{1}{8}$ in. deep. But as the crack depth increases, the crack becomes detectable. For example, for 3 in. of steel with a focal distance of 12 in., a crack 0.003 in. wide and $\frac{1}{8}$ in. deep is not detectable, but when the crack depth has increased to $\frac{1}{4}$ in., the crack is detectable. However, according to Norton and Ziegler,[5] the $\frac{1}{8}$-in. crack should be detected in 3 in. of steel. Hence we must conclude that the results of Norton and Ziegler must be qualified to permit consideration for crack width. Again referring to the same graph for steel 3 in. thick, we see that a crack $\frac{1}{8}$ in. deep is detectable when it is 0.005 in. wide, but not detectable when 0.004 in. wide. Here the ratio of flaw depth to total depth of steel remains constant, so the reason for the vanishing of the image of the flaw lies in the geometric effect.

SUMMARY

While these values hold for the focal distances used, it should be realized that increasing the focal distance will conceivably result in slightly lower limits of sensitivity.

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[5] J. T. Norton and A. Ziegler, "Sensitivity of the Gamma-Ray Method," *Transactions*, Am. Soc. Steel Treating, Vol. 22, p. 271 (1933).

DISCUSSION

Mr. Myron Zucker.[1]—In practice, cracks are usually irregular in shape, and seldom aligned with the X-ray beam. What conclusions can be drawn from the Doan-Sharp study as to the visibility of such cracks?

Mr. H. H. Lester.[2]—No conclusions can be drawn because this radiation would be parallel with the plane of the synthetic crack. It should be emphasized that where one is dealing with a synthetic crack, the conditions are not the same as those encountered where real cracks are dealt with. The results may have some points of similarity but should be used with caution.

Mr. H. F. Kaiser.[3]—In regard to the question brought up by Mr. Zucker, I should like to mention that Berthold[4] has worked with a special steel test block containing a cylindrical steel mandrel which carried slots of various widths and which could be turned to any desired angle. He found that if the angle between the slot and the direction of the X-ray beam was made as much as 15 deg., the minimum width of slot visible was increased by a factor of about five.

Mr. Gilbert E. Doan[5] (*Author's closure by letter*).—The limits found here are actual, quantitative limits. In so far as a natural crack departs from the quantitative conditions specified here, its detectability diminishes. No quantitative treatment can be given to the usual natural crack. It is however misleading to think that no conclusions about the *limit* of detectability of natural cracks can be drawn from these findings.

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[1] Engineering Division, The Detroit Edison Co., Detroit, Mich.

[2] Senior Physicist, Watertown Arsenal, Watertown, Mass.

[3] Assistant Physicist, Naval Research Laboratory, Washington, D. C.

[4] R. Berthold, "Defect Detecting in X-radiography, Part V. Detection of Cracks and Lack of Fusion," *Archiv für technisches Messen*, No. 98, August, 1939, p. 104.

[5] Professor of Metallurgy, Lehigh University, Bethlehem, Pa.

SYMPOSIUM ON PROBLEMS IN THE CLASSIFICATION OF NATURAL WATER INTENDED FOR INDUSTRIAL USE

INTRODUCTION

By F. N. Speller[1]

The papers and discussions in this symposium cover problems in reporting and interpreting the results of industrial water analyses and classification of natural waters, particularly with reference to their corrosivity in the temperature range from normal to slightly above the atmospheric boiling point. It should be emphasized that the symposium relates to "natural" waters. It is not necessary to point out that corrosion in water can often be greatly reduced by chemical treatment or by cathodic protection when these treatments are maintained under proper control. Furthermore, treatment of the environment (in this case natural water) is in many cases the most economical solution of corrosion problems.

Recently my attention was called to a rather irrational proposal to classify the corrosivity of natural waters according to their geographical location. A more rational procedure and formula has been developed to express the ability of natural waters to form protective deposits in metal pipes. This gives a corrosion index that conforms fairly well with experience in certain localities. However, it is well known that corrosion by water in closed systems within the above-mentioned temperature range is subject to many variables, and hence the problem of interpreting analyses and establishing even an approximate index of corrosion under these conditions is obviously a very complicated one. The ideal water corrosion index or formula should include all the more important factors, but any formula of this kind should be checked by quantitative service tests in typical waters and its limitations determined before it is advocated for general use. It is quite possible that a direct measure of the rate of attack under standardized conditions will be developed that may prove more reliable than any formula.

Soils are much more complicated in their physical and chemical properties than natural waters, and yet a fairly rational system of soil classification has been devised,[2] and their relative corrosivity determined in some cases, it is claimed, by a quick electrolytic laboratory test with an accuracy of about 70 per cent.[3]

Atmospheres have been roughly classified with respect to corrosion of metals as urban, rural, industrial, and marine, and the constituents that distinguish each group are fairly well understood but not defined. Therefore it does not seem unreasonable to attempt to obtain an approximate appraisal of the characteristics of waters supplied for industrial use.

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[1] Consulting Metallurgical Engineer, Pittsburgh, Pa.

[2] Scott Ewing, "Soil Corrosion and Pipe Line Protection," Am. Gas Assn., New York, N. Y. (1938).
C. F. Marbut, "Soils of the United States," Government Printing Office.

[3] Irving A. Denison and Robert B. Darnielle, "Correlation of an Electrolytic Corrosion Test with the Actual Corrosiveness of Soils," *Journal of Research*, Nat. Bureau Standards, Vol. 21, No. 6, December, 1938, p. 819 (*Research Paper 1157*).

Corrosion testing is now based on a better understanding of the causes and accelerating factors and much useful experience is available. The principles of testing were discussed in detail in the 1937 A.S.T.M. Symposium on Corrosion Testing Procedures.[4] These principles and methods of testing can be readily applied to the testing of variable environments such as natural waters. In this case a standardized lot of steel, and perhaps a few other commonly used metals, would be tested preferably under service conditions in typical waters. To insure comparative results by such tests it seems that only the main controlling factors, such as dissolved oxygen, carbon dioxide, temperature, and rate of flow need be included, without many of the accelerating influences that often occur in service, such as differential aeration, differential velocity of flow, the effect of contacts between dissimilar metals, the influence of anaerobic bacteria, and stress. Obviously, all these electro-chemical and biological effects cannot be covered in one formula, so while such a formula may be useful in many cases as a general guide, the user should be warned as to its limitations. Localized attack (pitting) is often more important than over-all corrosion (loss in weight).

These few details of the problems before us in this Symposium are mentioned only to illustrate their complicated nature. The papers and discussions which follow cover the subject in more detail and should be of considerable assistance to Committee D-19 on Water for Industrial Uses, in following up these subjects.

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[4] Symposium on Corrosion Testing Procedures, Chicago Regional Meeting; Continued at Fortieth Annual Meeting, Am. Soc. Testing Mats. (1937). (Available as a separate publication.)

REPORTING THE RESULTS OF WATER ANALYSIS[1]

By Robert C. Adams[2]

In all fields of man's effort there is a continuing need for improvement in accuracy and in convenience of communication. Whether the means of communication be the general language of a nation, that of mathematics, or the specialized vocabulary of a small group, what is said or written by one must be understood by another. Without this means for communication of one man's discovery to another, progress must labor under the necessity of continuous rediscovery of known facts instead of being able to build on the structure already erected.

In the field of water treatment there is no common language in which the results of water analyses can be reported. Within small groups mutual understanding has been achieved by definition and standardization of the terms used, but when a member of such a group speaks to a larger audience he must either restate his definitions or trust that his audience can translate for themselves with the aid of a dictionary of conversion factors. There is thus a recognized need for a system of reporting the results of water analysis which all can understand and use. The adoption of such a system would be an aid to progress.

It would be insufficient, however, to make understandability the sole criterion for selection of the system to be adopted. It should also be convenient to use; it should provide the means for simple calculation; and, most important in a scientific method, it must be precise. The analyst must be able to report what he actually finds—what really is in the sample, not hypothetical similarities.

There are several ways in which a system might be selected, developed, and attain eventual recognition and adoption. The heterogeneous systems now used might be tabulated and a plebiscite of interested chemists and engineers arranged to make a choice. There are at least two objections to this method: first, that it is improbable that any one system would attain a clear majority; and, second, that the approval of a majority is not infallible proof of the correctness of scientific truth.

A second method might be the delegation of a selected man or group of men to prepare an ideal system to be imposed upon all. Even if such a Utopian system could be developed, it is doubtful that its establishment would be successful. The decimal system of arithmetical notation, using nine digits and zero, is decidedly better than Roman numerals and an abacus. A duodecimal system, with eleven digits and zero, would simplify and reduce the time necessary for arithmetic calculation, but it is doubtful whether this further improvement will be adopted, although serious proposals to that end have been made.[3] England clings to an awkward monetary system, and her people expend a continuous added effort rather than change to a simple decimal coinage. It thus appears that a system of reporting water analyses must have familiar elements and involve a minimum of change in existing records in order to have promise of general adoption.

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[1] The opinions expressed in this paper are those of the writer and are not to be construed as official opinions of the Experiment Station or of the Navy Dept.

[2] Chairman, Subcommittee V, Committee D-19, on Classification; Chemical Engineer, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[3] "Calcolo decidozzinale del Barone Silvio Ferrari," Turin (1854) (quarto).

The third method of selection, and the most promising of success, appears to be the selection, by representatives of many fields, of the units of measurement now used which seem best adapted to use in all fields. Although it is improbable that such a system of reporting would be exactly the same as previously used by any one group, the chosen system would retain enough of the old means of reporting that the extent of the required revision of existing records would be minimized.

This third method, namely, selection by various representatives, was the method followed by a committee of this Society[4] in its two-year effort to reach an agreement on a method of reporting the results of water analyses capable of adaptation by all users of water. Bearing in mind the necessary requirements of familiarity, understandability, convenience, and precision, a standard method has been prepared which has been approved by the Society's Committee D-19 on Water for Industrial Uses and which is now being submitted to the Society for publication as a tentative standard.

According to the proposed method, the concentration of materials dissolved in water will be reported in either or both of two units: parts per million (ppm.) and equivalents per million (epm.). A part per million equals one milligram per kilogram of sample, or one pound per million pounds of water. In such dilute solutions as ordinary industrial waters, it is indistinguishable from one milligram per liter, but in reporting the analysis of brines, reagent solutions, or other concentrated solutions, the specific gravity of the sample must be considered. An equivalent per million is a gram equivalent weight per million grams of solution. Both are accurate weight per weight units, not dependent on the specific gravity of the solution. Parts per million can be converted to equivalents per million by dividing, and equivalents per million to parts per million by multiplying, by the equivalent weight of the ion or compound determined.

Parts per million is a convenient unit for measuring the gross amount of material dissolved or suspended in a water sample, or for measurement of the constituents of this total. It is an awkward unit to use for calculation of dosages or the products of reaction, because the molecular weight or an even fraction of the molecular weight of each constituent must be used in each portion of the calculation. By converting all ionic concentrations to equivalents per million, the reaction calculations are simplified to addition and subtraction. Volumetric procedures may yield direct results in equivalents per million. The acid titration of a sample usually does not identify the alkaline anions until the remainder of the analysis, including the pH, is known. However, the alkalinity in equivalents per million can be reported with accuracy.

The need for such a unit as equivalents per million is evidenced by the substitutes which have been used. Foulk[5] reports German chemists as using equivalent units in water analysis as early as 1865. Stabler's[6] reaction coefficient, the reciprocal of the equivalent weight, and reaction value, another name for equivalents per million, certainly were among the earliest uses of equivalent units in this country. The generally used method of reporting results as parts per million or grains per gallon of equivalent calcium carbonate is an unwieldy and arbitrary effort to achieve the simplicity in calculation offered by equivalents per million. Calcium carbonate was chosen as the specimen salt because of both its omnipresence in natural waters and its convenient molecular weight of 100 (equivalent weight of 50). Such a devious method leads to such absurdities as the reporting of chloride from condenser leakage as equivalent calcium carbonate, when there is no resemblance in chemical properties between the two.

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[4] Subcommittee V on Classification, of A.S.T.M. Committee D-19 on Water for Industrial Uses.

[5] C. W. Foulk, "Industrial Water Supplies of Ohio," *Bulletin 29*, Geological Survey of Ohio, Fourth Series (1925).

[6] *Water Supply Paper 274*, U. S. Geological Survey (1911).

Invention of the convenient term, equivalents per million, has been credited to Robb by Hall who first published the term in 1927.[7] The U. S. Geological Survey abandoned Stabler's reaction value, which is meaningless without definition, in favor of the precise but unwieldy term, milligram equivalents per kilogram.[8] It is probable that the experience of many others has been similar to that of the author, who formerly used equivalents per liter, abandoned that for equivalents per million in order to move the decimal point to a more convenient location, and later discovered that other chemists and engineers were using the same unit. The unit is so convenient that its simultaneous or successive discovery by many workers is not surprising.

Before proceeding to demonstration of the proposed method of reporting analyses, the widespread practice of reporting molecular combinations should be mentioned. This practice is undesirable because such a report is not an accurate representation of the analyzed sample. In solution all salts are dissociated into ions; analytical procedures determine either individual ions or groups of ions of similar electrical sign, so that if the analyst reports what he actually determines, he should report ions. A report of molecular combinations must be calculated from concentrations of the ions, and the nature of the report will be influenced by the opinions of the calculator, so that reports of analysis of identical samples by different laboratories might not be recognized as the same. A par-

TABLE I.

Constituent	Symbol	In Terms of	ppm.
Total hardness	TH	$CaCO_3$	120
Calcium	CaH	$CaCO_3$	83
Magnesium	MgH	$CaCO_3$	37
Alkalinity to methyl orange	Alk. A	$CaCO_3$	110
Basicity to phenolphthalein	Alk. B	$CaCO_3$	0
Sodium alkalinity	Alk. Na	Na_2CO_3	0
Free CO_2	CO_2	CO_2	6
Chlorides	..	NaCl	11
Sulfates	..	Na_2SO_4	28
Chlorides	..	$CaCO_3$	10
Sulfates	..	$CaCO_3$	20
Total cations	TC	$CaCO_3$	140
Theoretical mineral acidity	ThMA	$CaCO_3$	30
Free mineral acidity	FMA	$CaCO_3$	0
Silica	SiO_2	SiO_2	8
Total solids	TS	...	149

TABLE II.

Constituent	epm.	ppm.
Anions:		
HCO_3^-	2.20	134
CO_3^{--}	0	0
OH^-	0	0
Cl^-	0.19	7
SO_4^{--}	0.39	19
Total	2.78	
Cations:		
Ca^{++}	1.66	33
Mg^{++}	0.74	9
Na^+ (by difference)	0.38	9
Total	2.78	
Silica		8
Dissolved salts		211
Free CO_2		6

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[7] J. A. Robb and R. E. Hall, "A Physico-Chemical Study of Scale Formation and Boiler-Water Conditioning," *Bulletin 24*, Mining and Metallurgical Investigations, Carnegie Institute of Technology (1927).

[8] W. D. Collins, "Graphic Representation of Water Analyses," *Industrial and Engineering Chemistry*, Vol. 15, p. 394 (1923).

ticularly undesirable feature of this method of reporting is the hypothetical determination of nonexistent compounds, such as calcium bicarbonate.

For comparison, a water analysis from a published paper[9] is shown in Table I and the same data, in ppm. and epm. units, is given in Table II. The report given in Table I is repetitive, since some of the items are derived from others; and it is incomplete, since no value is given for sodium. The "alkalinity to methyl orange" in Table I is converted to HCO_3^- in Table II. A more accurate representation requires a more accurate method of determination[10] and becomes a problem of analysis and not one of reporting.

In the absence of a direct determination of sodium, the value given in Table II is the difference between the sums of the anions and cations determined. All other errors in analysis are combined in this one value. The value for free carbon dioxide in Table I is accepted in Table II, although it again is related to the bicarbonate and carbonate concentrations and to pH.[10] It is interesting to note that the figure for "dissolved salts" of Table II, obtained by totaling the concentrations of individual ions, is over 40 per cent greater than the similar figure for "total solids" in Table I.

The illustrative data of Table I originally were used[9] for the calculation of composition of that water after various treatments. Similar calculation can be made almost by inspection of the data of Table II. For example: after softening in salt-regenerated Zeolite, essentially all of the calcium and magnesium will be replaced by sodium; the anions remain unchanged. The effluent from the softener will therefore have 2.78 epm. of Na^+ and no other cations. The increase in dissolved salts will amount to:

$$(2.78 \times 23) - 51 = 13 \text{ ppm.}$$

The stoichiometric salt requirement for regeneration will equal the sum of calcium and magnesium ions or 2.4 epm. This value is converted to pounds of salt per million pounds of water by multiplication by the equivalent weight of salt, 58.46, to give 140. The regeneration efficiency is only about one third,[11] so that the actual salt requirement would approximate 420 lb. per million pounds of water.

Calculation of phosphate requirement for direct treatment of the water of Table II as boiler feedwater can be made almost as easily. Assuming that both magnesium and calcium are to be precipitated as phosphates, the phosphate requirement would be 2.4 epm. If at the same time it were desired to neutralize the feedwater alkalinity, it would be necessary to add 2.2 epm. of hydrogen ion. This could be done by the use of acid phosphates, a mixture of phosphoric acid and either monosodium or disodium phosphate. If phosphoric acid and monosodium phosphate were used, their proportions would be calculated as follows:

Let X = proportion of phosphoric acid, and

1 − X = proportion of monosodium phosphate:

$$\begin{aligned} 2.4X + 2.4(2/3(1 - X)) &= 2.2 \\ 2.4X \quad - \quad 1.6X &= 2.2 - 1.6 \\ 0.8X &= 0.6 \\ X &= 0.75 \end{aligned}$$

Acid requirement

$= 0.75 \times 2.4 = 1.8$ epm.

Monosodium phosphate requirement

$= 0.25 \times 2.4 = 0.6$ epm.

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[9] Howard L. Tiger, "Carbonaceous Zeolites—an Advance in Boiler-Feedwater Conditioning," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 60, p. 322 (1938).

[10] Tentative Method for Determination of Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Waters (D 513 - 38 T), 1939 Book of A.S.T.M. Standards, Part III, p. 1108.

[11] P. G. Bird, "Removal of Dissolved Salts from Water by Exchange Filters," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 635 (1938).

1.8 epm. of PO_4^{---} as 75 per cent phosphoric acid equals:

$\frac{1.8 \times 32.68}{0.75} =$ 78 ppm. (pounds per million pounds).

0.6 epm. of monosodium phosphate = $0.6 \times 46.01 = 28$ ppm. (pounds per million pounds).

If the magnesium will precipitate as hydroxide, and phosphate will be required only for calcium precipitation and alkalinity reduction, the calculation will be somewhat different. Magnesium precipitation will reduce the alkalinity from 2.2 epm. to 1.46 epm. The phosphate dosage must be chosen to provide 1.66 epm. of PO_4^{---} and 1.46 epm. of H^+. Since 1.46 is greater than two thirds of 1.66, phosphoric acid still must be used, and if it is used in conjunction with anhydrous disodium phosphate, the calculations would be as follows:

Let X = proportion of phosphoric acid, and

1 − X = proportion of disodium phosphate:

$$1.66X + \frac{1.66}{3}(1 - X) = 1.46$$

$$1.66X - 0.55X = 1.46 - 0.55$$

$$1.11X = 0.91$$

$$X = 0.82$$

Acid requirement

$= 0.82 \times 1.66 = 1.36$ epm.

Disodium phosphate requirement

$= 0.18 \times 1.66 = 0.30$ epm.

1.36 epm. of 75 per cent phosphoric acid equals:

$\frac{1.36 \times 32.68}{0.75} =$ 59 ppm. (pounds per million pounds).

0.30 epm. of disodium phosphate = $0.30 \times 47.37 = 14$ ppm. (pounds per million pounds).

The author and his committee believe that the proposed system of reporting water analyses meets the requirements of understandability, familiarity, convenience, and precision necessary to make it generally acceptable and usable. After adoption as a tentative standard of the Society, it will be subject to modification and improvement at the suggestion of all members. If those who make and use water analyses will make a sincere effort to employ the proposed system, particularly in publication, much time now lost in conversion calculations can be saved and we shall have made another step forward toward complete mutual understanding.

DISCUSSION

Mr. Howard L. Tiger[1] (*presented in written form*).—Mr. Adams and the Subcommittee on Classification of the Society's Committee D-19 on Water for Industrial Uses are to be commended for their efforts to clarify and simplify the cumbersome and confusing terminology that has been so frequently used in reporting water analyses. There are only a few comments that I should like to make on the suggested procedure for reporting such analyses.

There is surely no basis for reporting the dissolved substances in water as hypothetical compounds, and, as Mr. Adams points out, the logical method is to report the analysis in terms of the individual anions and cations, as this is the form in which the substances actually exist in dilute solution. It should be borne in mind, however, that in addition to presenting these basic figures, it is desirable to present some additional figures for the convenience of those who will have to interpret the analyses and who may not be so familiar with the methods of making calculations from the equivalents. For example, the reader of an industrial analysis usually wants to know the total hardness, and, in the case of boiler feedwater, he usually wants to know the sulfates and the sodium alkalinity in terms of Na_2SO_4 and Na_2CO_3, respectively, so that he may readily determine whether the composition of the boiler feedwater complies with the sulfate:carbonate recommendations of the American Society of Mechanical Engineers. Furthermore where the new carbonaceous hydrogen ion exchange materials are under consideration, it is desirable to summarize figures on theoretical mineral acidity, etc.

The need for this additional information in convenient form explains why these items were included in Table I of Mr. Adams' paper which, as he states, is a copy of a table in my paper, "Carbonaceous Zeolites—an Advance in Boiler-Feedwater Conditioning," published in the A.S.M.E. *Transactions* for May, 1938, pages 315–325. Mr. Adams calls these items repetitive, but in my opinion it is desirable to supplement the simplified form recommended in his Table II with such additional figures as may be found convenient for general industrial use.

Another point which Mr. Adams brings out in his paper is the total solids calculation in his Table II as compared with the figure given in Table I. In Mr. Adams' calculation, he considers the bicarbonates to be present in that form when he makes his total solids calculation. Actually, the principal significance of the total solids calculation is for boiler feedwater purposes, and it will be noted that Table I was presented in a paper relating to boiler feedwater. From that point of view, one is interested in the total solids that will exist in the concentrated boiler salines.

It is well known that all the bicarbonates and the bulk of the carbonates decompose in the boiler, so that the concentrated salines contain a mixture of hydroxides and carbonates—the higher the pressure, the higher the proportion of hydroxides. It appears logical, therefore (at least from the boiler feedwater point of view), to calculate the total solids on the basis of the compounds that will exist in the boiler. It is the lower molecular weight of these compounds which accounts for the difference between Mr. Adams' calculation of 211 ppm. as against my previous calculation of 149 ppm. total solids. In this connection, it might be advisable to indicate on the analysis that the calculation of total solids is based on the conditions that prevail in the boiler.

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[1] Vice-President, The Permutit Co., New York, N. Y.

I agree also with Mr. Adams' suggestion to report the results in terms of equivalents, in order to simplify various calculations in connection with an analysis. However, during the transitional period required to familiarize engineers and industrial men with this simplified method of expression, it will undoubtedly be advisable also to present the results in terms of ppm. And in presenting the results in that converted form, I think it would be better to express the results in terms of ppm. as $CaCO_3$.

As to the form of expression for the equivalents, Mr. Adams states that he formerly used equivalents per liter, but that he abandoned that "for equivalents per million in order to move the decimal point to a more convenient location. . ."

In this connection, it may be pointed out that the same result may be obtained by reporting the analysis in milliequivalents per liter (meq. per l.). This moves the decimal point to the right, and at the same time it retains certain definite advantages which are inherent in milliequivalents per liter as compared with the suggested term "epm.":

1. "epm." is vague and indefinite, as it is not clear whether the term means equivalents per million equivalents of water or gram equivalents per million grams;
2. "epm." is not related to the actule method of measurement used in tha laboratory where one analyzes the constituents in a certain volume of water;
3. "epm." is not an accepted chemical term like "meq. per l." The latter fits in with the international language of the chemical laboratory, and it permits simple conversion to milligrams per liter which is a universal term for any water analysis and which, furthermore, is synonymous with "ppm."

MR. J. D. YODER[2] (*by letter*).—Very often in the consideration of water treatment especially where several steps of treatment are applied it is desirable to show in parallel columns the effect of each proposed treatment.

TABLE I.—OPERATING RESULTS SHEET.[a]

	No. 1		No. 2, epm./50	No. 3, epm./50	No. 4, epm./50
	ppm.	epm./50			
Ca	1.4	3.5	3.5	1.0	...
Mg	0.7	2.9	2.9	1.0	...
Na	0.9	1.9	9.9	16.3	815
CO_2	...	...	...	...	...
HCO_3	1.2	1.0	5.0	5.0	...
CO_3	...	...	...	...	40
OH	...	...	...	...	160
PO_4	...	...	...	2.0	50
Cl	1.2	1.7	1.7	1.7	85
SO_4	5.4	5.6	9.6	9.6	480
	ppm.				
SiO_2	2.2	2.2	2.2	2.2	110
R_2O_3	0.3	0.3	0.3	0.3	...
TDS		8.3	16.3	18.3	815
Turbidity	2.4	2.4	0.0	0.0	115
pH	(5.8)				

[a] No. 1 = Raw water—pond SW-4, 10/6/38.
No. 2 = Coagulated and filtered—(alum + Na_2CO_3).
No. 3 = Zeo-Karb-Na treatment—plus Na_3PO_4 (in boiler).
No. 4 = No. 3 concentrated 50 times in boiler.

The use of "epm." generally requires fractional units which are inconvenient and are not closely related to the average engineer's conception of the hardness or mineral solids in the water. Accordingly, I am suggesting for consideration the use of the term "epm./50." The Operating Results Sheet (accompanying Table I) shows how these units have been applied.

To employ epm./50 is the same as

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[2] Manager of Sales, Boiler Feedwater Division, The Permutit Co., New York, N. Y.

reporting the ions in terms of calcium carbonate but the term epm./50 is more convenient and is very similar to what Mr. Adams' committee has already recommended.

When dealing with highly concentrated solutions the term epm. would be more convenient but for water analyses the smaller units are preferable. For convenience the total dissolved solids expressed in ppm. might be considered the sum of the cations in epm./50 plus SiO_2 in solution. This of course is not strictly correct but it serves all practical requirements. The hardness in ppm. calcium carbonate is the sum of the calcium and magnesium epm./50.

I should be glad to have Mr. Adams' comments.

MR. R. C. BARDWELL.[3]—I am a member of Mr. Adams' committee and do not want to disagree too strongly with the excellent work that has been done by Mr. Adams. It has been hard and tedious work that he has performed. But the fact still remains that, as far as boiler water analyses are concerned, I would estimate that at least 80 per cent are not being reported in the terms he has recommended.

It is a fine thing for chemists to report exactly what they find in the form that they find it. A chemist who makes a water analysis conscientiously and properly does just about what Mr. Adams has outlined in his report. However, when it comes to making the report of the analysis, it has to be put in a form understandable to the persons to whom the report is made.

As far as boiler water analyses are concerned, the general practice has been practically uniform to report the water analysis in the form of hypothetical combinations. To a chemist or research worker, this may not be exact or scientifically proper, but this form has been so generally used that it appears to be understandable to the persons who require this information. The widespread use of this form entitles it to some consideration.

In my opinion, the most helpful action that can be taken by this Society would be to agree on a standard method for making these hypothetical combinations where such form of report is desired. Regardless of the action taken by the Society, the practice of using hypothetical combinations in making reports of boiler water analyses will be continued. It would promote uniformity if these hypothetical combinations are made in accordance with a standard method such as has been recognized and adopted by other technical societies who have investigated the problem. The American Railway Engineering Assn. in 1923 adopted a recommended plan for hypothetical combinations in water analysis reports; the American Water Works Assn. adopted a similar plan in 1929.

If water analysis reports are for high technicians or research work, there is no question but that the ionic method as outlined is a good form. If the report is to be made in a form which will be understood by persons responsible for its application in the industrial field, this will not apply, and, in my opinion, we should conform to the plan that has proven successful and is being universally used. This form is a standardized method for hypothetical combinations.

It is appreciated that this recommendation is against the vote of the committee and is a minority report, but I still think it proper.

MR. SAM S. TAYLOR[4] (*by letter*).—The following comment is based on Mr. Adams' paper.

The literature describes many diverse ways of reporting the results of water analyses. Each method used has been advanced to an established practice by groups who represent various schools of thought and who understand the analysis reported by the method of their preference. Some municipal water plant operators are interested mainly in the hardness of the water, and to these men water analyses are reported in terms equivalent to calcium carbonate (calcium carbonate usually is the main cause of hardness). Another group desires the results of water analysis reported as salinity and alkalinity. Some desire the results shown as hypothetical combinations. Some desire only the ionic statement of soluble components. Analyses are reported variously as parts per million, milligrams per kilogram, milligrams per liter, grains per U. S. gallon, and (in foreign countries) grains per imperial gallon and parts per 100,000; in all countries individual variations are used. All of these methods of reporting water analyses, used singly or in combinations, have the advantage of being understood by those for whom the analyses were made.

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[3] Superintendent of Water Supply, Chesapeake & Ohio Railway, Richmond, Va.

[4] Assistant Petroleum Engineer, Petroleum Equipment Station, Bureau of Mines, Bartlesville, Okla.

TABLE II.—AN OIL-FIELD BRINE ANALYSIS CALCULATED FOR REPORTING BY TWO METHODS.

Radical	Bureau of Mines Method		Method Suggested by Adams	
	Parts per Million (milligrams per liter)	Reacting Values in Percentage (Palmer)	Parts per Million (milligrams per kilogram)	Equivalents per Million (reaction value)
Iron (Fe)	31	0.03	29	1.04
Calcium (Ca)	5 134	6.06	4 723	235.68
Magnesium (Mg)	1 546	3.01	1 422	116.94
Sodium (Na)	39 772	40.90	36 607	1591.83
				1945.49
Bicarbonate (HCO_3)	67	0.03	62	1.02
Sulfate (SO_4)	103	0.05	95	1.98
Chloride (Cl)	74 820	49.89	68 832	1941.28
Bromide (Br)	104	0.03	96	1.20
Iodide (I)	1	......	1	0.01
				1945.49
Total Solids	121 578	100.00	111 867	
Specific gravity at 15.6 C. (60 F.)	1.087		1.087	

Water analysts in this country use similar methods of procedure, and a large number have adopted procedures or modifications thereof, as outlined by the American Public Health Assn. The method used by the chemist in reporting the analyses is dictated by the group that is to use the analyses; naturally the method used is one understandable by the majority of the group.

If water analyses were reported universally by giving the ionic concentration of each component determined in milligrams per liter and the specific gravity of the sample, any type of a reported statement could be calculated, and the result would conform with a large number of the many analyses already published. Fortunately many organizations report water analyses by at least two methods, including parts per million (milligrams per liter) as do the Bureau of Mines, Geological Survey, and many State Boards of Health.

The Bureau of Mines has given the subject of reporting the results of water analyses careful consideration and has recorded its conclusions in *Technical Paper 432*. In that paper it has recommended "that the use of 'milligrams per liter' be continued to designate parts per million, while the term 'parts per million by weight' is reserved to designate milligrams per thousand grams of sample."

The proposals of Mr. Adams are correct fundamentally, but they present nothing new. Furthermore, the standard method of reporting the results of water analysis adopted by the American Public Health Assn. is the same as the method proposed by Mr. Adams, but its method of reporting is quite liberal in that it recognizes the advantage of reporting results understandable by groups adhering to various methods of reporting and comparable with those published in the literature.

The accompanying Table II shows the manner of reporting the results of a brine analysis by two methods. Columns 2 and 3 indicate how the Bureau of Mines reports (in column 2 as milligrams per liter) the analysis, and in columns 4 and 5 the same analysis is shown by the method of reporting suggested by Mr. Adams. The values shown in column 4 are defined in *Technical Paper 432* as milligrams per thousand grams of sample or "parts per million by weight." These values were obtained by dividing milligrams per liter (ppm. by volume) by the specific gravity of the sample at the same temperature. The Bureau in reporting its findings in milligrams per liter is conforming with customary practice in measuring water; in other words, water is measured by volume rather than by weight. The values shown in columns 4 and 5 were calculated from the values shown in column 2. The Bureau's analysis can be recalculated into any desired scheme of reporting.

MR. ROBERT C. ADAMS[5] (*Author's Closure*).—As Mr. Taylor says, no new proposals have been offered in the paper. What it does propose is the establishment of a practical, usable basis for uniformly understandable reports of water analyses by reasoned selection from what is old. The outline which has been presented can be elaborated and extended for special applications without loss of this basic soundness and understandability.

Mr. Tiger would provide certain combinations of ions, such as *Theoretical Mineral Acidity*, in his report of analysis. There can be no objection to this, nor to his estimation of residue on evaporation in a boiler, so long as these special combinations are defined and are not given names otherwise defined in the standard method of reporting. The writer believes the term *epm.* is as widely used and understood as Mr. Tiger's proposed alternate *meq. per l.* and has the advantages of brevity, homogeneity (being a weight-per-weight unit), and analogy with ppm. with which it is associated.

The use of convenient multiples of the basic units, suggested by Mr. Yoder, should extend the usefulness of the standard. Whether epm. should be multiplied by 10, 50, 100 or another number is immaterial so long as the multiplier is clearly stated. However it would seem that when the concentration is multiplied by 50, for example, it should be expressed by *epm.* × *50*.

Mr. Bardwell's assistance as the spokesman for the opposition has been appreciated by the Committee and the writer. His further cooperation in extending his private system, which probably resembles the proposed standard, to more general use and recognition would also be helpful.

The general agreement in all the discussions on the soundness and accuracy of the proposed standard is heartening evidence that the Committee has cleared a usable meeting ground in the forest of water reporting. If the epm.-ppm. system can be maintained as a basis for the adaptations necessary for special cases we shall have progressed in mutual understanding.

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[5] Chairman, Subcommittee V, Committee D-19, on Classification; Chemical Engineer, U. S. Naval Engineering Experiment Station, Annapolis, Md.

A REVIEW OF DATA ON THE RELATIONSHIP OF CORROSIVITY OF WATER TO ITS CHEMICAL ANALYSIS

By V. V. Kendall[1]

At various times suggestions have been advanced to correlate the corrosivity of a water with its chemical analysis or some particular property of the water. In reviewing these various suggestions we have made no attempt at an exhaustive discussion. Our object is to stimulate further work in the development of formulas to make them broader in application and to bring to light data collected by various investigators that will assist in the eventual solution of the problem.

It is recognized that if we remove the air or oxygen from natural water, corrosion is eliminated. Also, if we maintain a sufficiently high pH in a water, corrosion is reduced to a minimum in spite of the oxygen content. (This statement does not apply to very elevated temperatures.) If the chemical constituents of a water are in such proportions that calcium carbonate is precipitated on the metal surface, then the water is noncorrosive. These and other facts have been used from time to time to forecast the corrosivity of a water or to form a basis for treatment to render it noncorrosive. The formulation of hypothetical combinations of the constituents of a water are legion. Assumptions as to the corrosivity of a water based upon such hypothetical combinations may be misleading. Complete analytical data should be given. If a particular hypothetical combination has been found to be useful in a local water where the properties of that water are known, by all means report it; but at the same time, give the complete ionic analysis. This permits later recalculations on the basis of more information and also permits other investigators with different ideas to utilize the data.

Stabler[2] formulated mathematically a comprehensive scheme for interpreting an industrial water analysis. His "coefficient of corrosion" represented the net concentration of hydrogen ion in the water and was the sum of any hydrogen ion in the water plus that resulting from the hydrolysis of aluminum, iron, and magnesium salts minus that removed by carbonates and bicarbonates. He recognized the effect of calcium or the precipitation of calcium carbonate. If the calcium content was sufficient to provide definite precipitation of calcium carbonate, the water was noncorrosive, but if a borderline condition prevailed it might or might not be corrosive.

The use of pH adjustment for the control of corrosion in municipal water supplies was developed in large part by the efforts of J. R. Baylis. The maintenance of a pH value on the alkaline side was found to prevent "red water" trouble. In addition to maintaining the pH at or above the calcium carbonate equilibrium, Baylis[3] had the further idea that "The problem in treatment of water to prevent corrosion is to produce conditions nearest ideal for the precipitation of all the products of corrosion at the point where corrosion is taking place—that is, where every tendency to corrode will quickly form insoluble compounds."

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[1] Corrosion Engineer, National Tube Co. (Subsidiary of U. S. Steel Corp.), Pittsburgh, Pa.

[2] Herman Stabler, "The Mineral Analysis of Water for Industrial Purposes and Its Interpretation by the Engineer," *Engineering News*, Vol. 60, pp. 355-357 (1908); also "Some Stream Waters of the Western United States," *Water Supply*, Paper 274, U. S. Geological Survey (1911).

Palmer[4] evolved a system of classification of ground waters characterized by division of the constituents into percentage amounts of primary and secondary salinities, and primary and secondary alkalinities. Primary and secondary salinities consist of the chlorides and sulfates of sodium and potassium and of calcium and magnesium, respectively. Primary and secondary alkalinities consist of the carbonates and bicarbonates of sodium and potassium and of calcium and magnesium, respectively. Tertiary salinity (acidity) is the excess (if any) of salinity over primary and secondary salinity. Mills[5] used the Palmer system of classification in a survey of corrosion in the oil and gas fields. His comparison of the chemical qualities of corrosive and noncorrosive oil- and gas-well waters is given in the following table:

Properties of Reaction	Corrosive, per cent	Nonocorrosive, per cent
Primary salinity	50 to 91	16 to 83
Secondary salinity	8 to 30	none
Primary alkalinity	0 to 2	14 to 82
Secondary alkalinity	0 to 14	0.8 to 6
Chloride salinity	7 to 100	16 to 99.7
Sulfate salinity	0.2 to 92.4	0.3 to 84
Concentration of saline matter, p.p.m.	2106 to 164 000	5000 to 12 000

In his summary he states "Field evidence indicates that corrosion in wells rarely occurs when the waters have a primary alkalinity exceeding 14 per cent. High concentration of chloride and sulfate salts, more especially calcium and magnesium chlorides, in the absence of primary alkalinity, is especially favorable to corrosion." Such a classification is more applicable to heavy brines or waters, such as are found in the oil and gas fields, and appears to be of value in that field.

In 1936, Langelier[6] introduced a mathematical evaluation of the ability of a water to precipitate calcium carbonate. If a water is unsaturated with respect to calcium carbonate, it will attack the metal. If it is oversaturated, a coating of calcium carbonate will be precipitated on the metal and will protect it. For the derivation of the equations, reference should be made to the original paper. For our purpose we are concerned only with final equations which follow:

$$\text{Corrosion Index} = \text{pH}_{\text{actual}} - \text{pH}_s$$

$$\text{pH}_s = (\text{pK}'_2 - \text{pK}'_s) + \text{pCa}^{++} + \text{pAlk.}$$

The two latter terms of the second equation are negative logarithms of the molal and equivalent concentrations of calcium and titratable base, respectively. pK'_2 and pK'_s are the negative logarithms of the second dissociation constant for carbonic acid and the activity product of the CaCO_3, respectively. The difference $(\text{pK}'_2 - \text{pK}'_s)$ varies with ionic strength (salinity) and temperature, a table of values for which is given. pH_s is a hypothetical value representing the pH at equilibrium *assuming no change in composition* of the water in bringing it to equilibrium. For rapid calculation of pH reference should be made to the monogram published by Hoover.[7]

If the corrosion index is zero, the

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[3] J. R. Baylis, "Factors Other than Dissolved Oxygen Influencing the Corrosion of Iron Pipes," *Industrial and Engineering Chemistry*, Vol. 18, p. 370 (1926).

[4] Chase Palmer, "The Geochemical Interpretation of Water Analysis," *Bulletin No. 479*, U. S. Geological Survey, 31 pp. (1911).

[5] R. Van A. Mills, "Protection of Oil and Gas Field Equipment Against Corrosion," *Bulletin No. 233*, U. S. Bureau of Mines, 127 pp. (1926).

[6] W. F. Langelier, "The Analytical Control of Anti-Corrosion Water Treatment," *Journal*, Am. Water Works Assn., Vol. 28, October, 1936, p. 1500.

[7] C. P. Hoover, "Practical Application of the Langelier Method," *Journal*, Am. Water Works Assn., Vol. 30, November, 1938, pp. 1802–1807.

water is in equilibrium at that temperature. A positive index indicates oversaturation and a tendency to crystallize or to lay down a protective coating of $CaCO_3$ in the pipe, and a negative index indicates undersaturation or a tendency to dissolve an existing carbonate coating. In addition to the data offered by Langelier, DeMartini[8] reported data from California cities and concluded that "in the case of cold waters that have a saturation index more positive than − 0.5, little corrosion difficulty is to be expected. For hot waters having an index more positive than zero, slight difficulty is to be expected." "*This should not be interpreted to mean that waters meeting this standard will be absolutely free from corrosion effects.* [Italics are Kendall's.] Pitting may continue, but at a much slower rate. It may also require considerable time for waters brought to the above standard to build up a protective coating on the metal."

The practical data given were based upon the occurrence of "red water" complaints. It is a known fact that corrosion can proceed without actual precipitation of iron in the water, or "red water," as it is called. Below the point of iron precipitation, corrosion is proceeding, but it becomes serious when the rate of corrosion is increased to the point of "red water." If the corrosion index is zero, that means that the water is in equilibrium with calcium carbonate but that no calcium carbonate is being precipitated and hence no protection is afforded the pipe. The ideal condition is the precipitation of a thin layer of calcium carbonate that is protective and is maintained at that thinness, but that does not build up to a thickness that decreases the rate of flow of the water. This means further that actually to protect the pipe, the corrosion index must be maintained at a positive value and not at zero or a − 0.5 value. If the corrosion index is very slightly positive the rate of precipitation of calcium carbonate may be so slow that it will offer very little or no protection to the pipe. Langelier, himself, states that "In those cities where pH adjustment is practised, either for corrosion or incrustation control, the tendency is to undertreat." The Langelier index will separate waters definitely corrosive or noncorrosive but will not give the actual picture in the range at or close to calcium carbonate equilibrium.

In dealing with municipal waters, our first problem is to determine a measure of the corrosivity of the water. General experience is unreliable and misleading. Laboratory tests are few and their reproduction of service conditions has been questioned. There remains the service test. To determine what data might be obtained from this source, records of service tests in hot water lines in various cities were investigated. In making a service test, steel and wrought iron pipes, 2 or 3 ft. long, are placed in the hot water line in alternate positions —for example, steel, wrought iron, steel, wrought iron, etc. Two or three pieces of each material are used. Other materials may be included. After the pipe has been in service from one to twelve years, it is removed, cleaned, and the depth of pitting measured. No attempt is made to determine the loss in weight, since practical conditions do not permit accurate loss in weight determinations, and the life of the material is dependent on pitting and not on loss in weight. Cleaning is generally by ammonium citrate, but wire brushing and shot blasting may be used. In recent tests, shot blasting was used as such cleaning is thorough and the depth of pitting is not appreciably affected. In measuring the depth of pitting, 6 in. on each side of the coupling are ignored to prevent any dissimilar metal effect from affecting the results. The average of the ten deepest pits on each piece of pipe is taken as the depth of pitting of that piece, and where several pieces of the same material are included, the average of these averages is taken as the depth of pitting of that material. The service test insures identical conditions of exposure for different materials and shows what may be expected from any material under the particular conditions encountered.

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[8] F. E. De Martini, "Corrosion and the Langelier Calcium Carbonate Saturation Index," *Journal*, Am. Water Works Assn., Vol. 30, January, 1938, pp. 85-111.

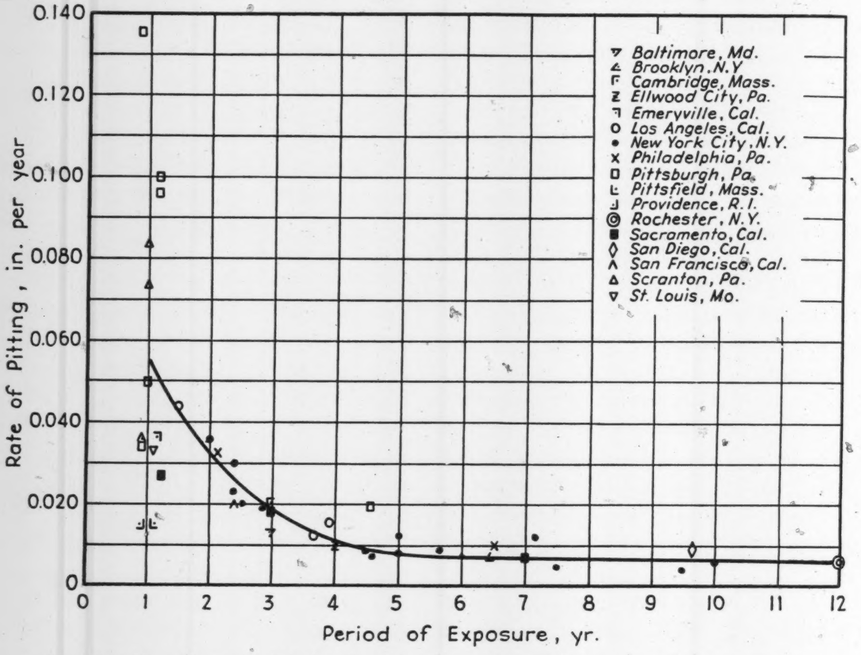

FIG. 1.—Data from 42 Service Tests in Hot Water Lines in Seventeen Cities.

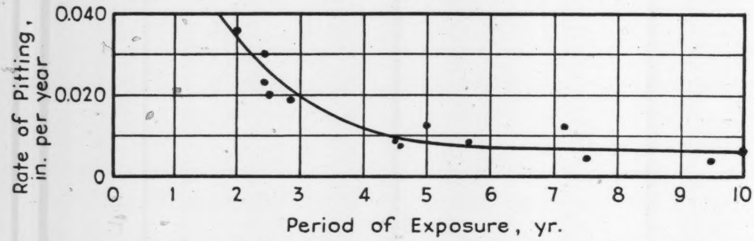

FIG. 2.—Service Tests in Hot Water Lines in New York, N. Y.

The records of service tests in hot water lines in various cities were then consulted and the rate of pitting plotted against period of exposure. These data, consisting of 42 service tests in 17 cities, are shown in Fig. 1. Experimental conditions such as temperature, velocity of flow, etc., varied from test to test, as would be expected. In spite of such variation, the points at 2-yr. exposure and longer fall on a definite curve. At approximately 1-yr. exposure, the variation is considerable. This would indicate that the initial attack on the metal, as measured by the results within the first year, is a function of the corrosivity of the water. After the initial period of rust formation, the rate of corrosion is slowed down. The attack on the metal is then dominated by the character of the rust, its porosity, its ability to permit diffusion of oxygen, etc. This decreasing rate of corrosion with time is further shown by plotting separately (Fig. 2) the results from 13 service tests in New York City. It is evident that the period of exposure must be taken into consideration in assigning a corrosion value to a water or in comparing the corrosivities of several waters.

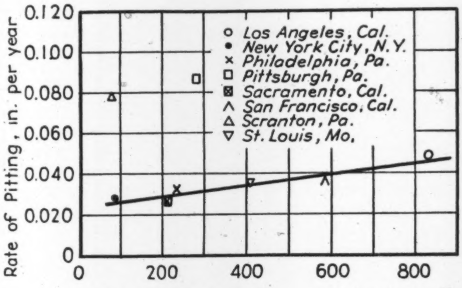

FIG. 3.—Effect of Conductivity of Water on Rate of Pitting.

The service tests at an exposure of approximately one year were then separated and the records were consulted to obtain the analyses of the waters during the period of the tests. This left 16 tests in eight cities for which water analyses were available. In some cases, the water analyses were not available for the exact test periods and the nearest analysis in point of time was then used. In general, the average yearly analysis varied little from year to year.

The Langelier corrosion index could not be calculated due to lack of pH data. Plotting the various constituents of the water and ratios of certain constituents to others resulted in "shotgun" diagrams.

If our theory of electrochemical corrosion is correct, there should be a relationship between the specific electrical conductivity and corrosion. Specific conductivity measurements have been used for determining the purity of steam, of supplies used for boiler-feed purposes, and the concentration of boiler and evaporator water.[9] The electrical conductivities of the water were calculated using the factors given by Hecht and McKinney for calculating specific conductance from the ionic analysis.

The points shown are averages as follows: New York City, 4 tests; Pittsburgh, 5 tests; Scranton, 2 tests; and one each for the rest. These data are shown in Fig. 3. Correlation between rate of corrosion and conductivity is shown in the data from six out of the eight cities. The service tests in Pittsburgh and Scranton gave a high rate of corrosion. The 5 tests in Pittsburgh varied from a rate of 0.03 in. per yr. to 0.13 in. per yr. The probable explanation of the high figures for Pittsburgh and Scranton is seasonal variation in the pH value of the water.

We believe that the correlation shown between rate of corrosion and specific electrical conductance warrants further investigation, particularly in view of the practical nature of the data and the broad averages for water analyses which it was necessary to use. It would be helpful if those who have conductivity equipment would include this determination in their water analyses and thus furnish more data on this point.

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[9] Max Hecht and D. S. McKinney, "Electrical Conductance Measurements of Water and Steam, and Applications in Steam Plants," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 53, No. 8, May–August (1931).

SUMMARY

In reviewing the data on the relationship of corrosivity of water to its chemical analysis, the author has not attempted an exhaustive discussion but has endeavored only to bring out the highlights of the problem and to stimulate discussion and interest with the hope that further data will be brought to light that will advance our quantitative knowledge of the subject.

In the use of service test data in obtaining a rate figure for the corrosivity of a water, consideration must be given to the period of exposure. The corrosive character of a water is best indicated in short-time periods before the retarding effect of the rust coating becomes predominant. Data from a large number of service tests in hot water indicate that after an initial period, the rate of corrosion is more or less constant.

Some correlation was shown between corrosivity and specific electrical conductivity in a number of service tests in different cities.

DISCUSSION

MR. F. L. LAQUE[1] (*presented in written form*).—It seems evident from Mr. Kendall's paper that it is not going to be easy to establish a consistent relationship between the corrosivity of water and its chemical analysis—*as usually reported.*

The following types of water might be considered as being noncorrosive:

1. Those able to protect any metal by depositing continuous impervious and permanent films, such as lime deposits, which originate wholly within the water and are maintained by the water.
2. Those able to protect certain metals by the formation of insoluble corrosion product films.
3. Those so constituted as to alkalinity or corrosion-inhibiting constituents as to be noncorrosive to certain metals even in the absence of films of the types covered by items 1 and 2.

All other waters might be considered as potentially corrosive, with the intensity of corrosion in any case being influenced by the usual factors of aeration, temperature, and velocity, as well as by its chemical composition, acidity, and electrical conductivity. The effects of chemical analysis of such waters will vary considerably from metal to metal and it does not seem likely that any generally applicable relationship between corrosivity and composition will be developed. This statement also applies to noncorrosive waters of types 2 and 3 mentioned previously.

It appears that most of the efforts to distinguish between corrosive and noncorrosive waters have been directed towards item 1 in the suggested classification of noncorrosive waters, and with particular reference to iron and steel.

Other estimates of the probable corrosivity of waters towards various metals and alloys must be based principally on practical experience and the limited amount of available corrosion test data. Efforts to correlate these practical experiences and test data with water composition have been made difficult by the lack of complete information on the chemical characteristics of the waters under consideration.

Fortunately, the chemical make-up and corrosivity of a very large part of the water that must be handled, that is, the water supply of the larger cities, remain fairly constant and are subject to continuous control. Furthermore, any major changes that may occur will probably be in the direction of lower corrosivity. Consequently, it may be unnecessary to know all about the composition of these waters in order to use past experience in a locality as a basis for providing for probable corrosion of metals in the future.

It seems reasonable to expect that, with a better understanding of what information on water composition is needed by the corrosion investigator, eventually a mass of data will become available that will enable engineers to examine the chemical characteristics of a new water supply and predict the probable behavior of the numerous metals and alloys which may be used in contact with it; or devise a method of treatment of the water which will reduce its corrosiveness towards the ordinary materials of construction. This may entail some changes in the present methods of determining and reporting the chemical characteristics of water, particularly with respect to such features as the content of dissolved gases, acidity and electrical conductivity.

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[1] Research and Development Division, The International Nickel Co., Inc., New York, N. Y.

TABLE I.—RESULTS OF TESTS ON STEEL SPECIMENS EXPOSED TO CORROSION BY TAP WATER.

Locality	Duration of Exposure, yr.	Corrosion Rate, in. per yr.		Corrosion Rate by Pitting from Kendall's Fig. 1, in. per yr.		Remarks
		From Weight Loss	By Pitting	From Curve	From Points Referring to Same Locality	
Long Branch, N. J.	2.5	0.002	>0.02	0.025	.....	Cold water (Avg. 56 F.) in treatment plant
Provincetown, Mass	0.3	0.003	0.01	.....	.....	Hot water tank
Manhattan, N. Y.	1.8	0.007	>0.028	.....	0.035	Hot water tank
Hollis, L. I.	1.2	0.008	>0.045	0.050	.....	Hot water tank
Camden, N. J.	0.6	0.008	0.041	.....	.....	Cold water
	1.7	0.006	>0.029	.....	.....	Cold water
	1.7	0.006	>0.029	0.037	.....	Hot water tank
Brooklyn, N. Y.	1.2	0.006	>0.043	0.050	.....	Hot water tank
Aurora, Ill.	1.2	0.002	0.014	0.050	.....	Hot water tank
Glen Head, L. I.	1.2	0.008	>0.041	0.050	.....	Hot water tank
Johnstown, Pa.	1.4	0.008				Hot water tank
Pittsburgh, Pa.	0.7	0.013	>0.07		0.05 to 0.1	Hot water tank
Jackson Heights, L. I.	1.6	0.006	>0.03	0.038	.....	Hot water tank
Kingston, N. Y.	0.5	0.018	>0.106	.....	.....	Hot water tank

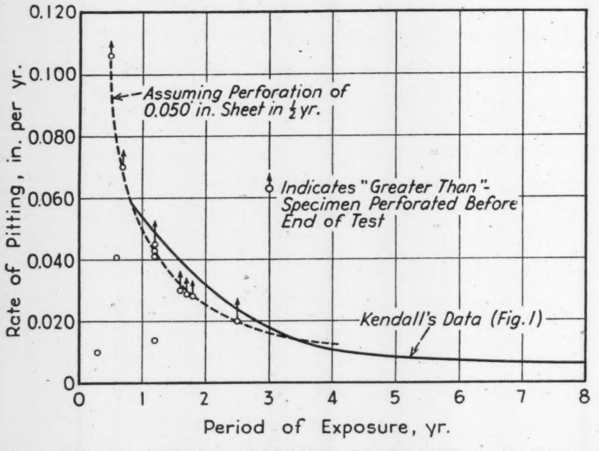

FIG. 1.—Comparison of Rate of Pitting of Mild Steel in Hot Water by Tests of Independent Investigators.

The data which Mr. Kendall has provided on the rates of penetration of steel pipe by hot water in several localities should be of considerable practical value to those concerned with the use of steel pipe in those localities even though he was unable to find any consistent relationship between pipe life and water composition.

During the past few years, opportunities have been provided for the inclusion of mild steel in corrosion tests carried out by exposing specimens in hot water tanks in several localities. The test specimens were supported on spool-type specimen holders[2] which were placed inside the hot water heating and storage tanks. The results of these tests are shown in the accompanying Table I and Fig. 1 which also include for convenient comparison the rate of pitting *versus* time of exposure data taken from Fig. 1 of Kendall's paper.

Unfortunately, the thickness of the steel specimens (0.05 in.) in these tests was not great enough to provide an accurate measure of the rate of pitting since, in most cases, the specimens were perforated during the test period. So far as pitting is concerned, the figures indicate only minimum rates. However, the positions of these minimum rate points with reference to the curve from Kendall's Fig. 1 may be considered as tending to support Kendall's observations.

The broken line curve indicates the rates of pitting which would bring about perforation of the 0.05 in. thick specimens at the various time intervals. A comparison of this curve with the minimum rate points from the corrosion tests suggests that in most of the waters studied the 0.05 in. thick specimens may have been perforated after the first six months of exposure.

Whatever value these data may have in relation to Kendall's figures, it is obvious that they support the conclusion that weight loss determinations provide no measure of the durability of steel in the waters under consideration. In addition, they may have some value in indicating the minimum rates of pitting of steel by hot tap water in the localities in which the tests were made.

Another point which seems worth mentioning is that the apparent similarity between Mr. Kendall's curve and the broken line curve in Fig. 1 of this discussion might be taken as a strong indication that Kendall's curve also satisfies the condition where most, if not all, of the observed pitting occurs within the early periods of exposure. It is suggested that if Mr. Kendall were to replot his data on the basis of total depth of pitting rather than rate of pitting *versus* time of exposure, the tendency for pitting to slow down or cease after a few years would be shown more clearly.

MR. W. F. LANGELIER[3] (*presented in written form*).—Mr. Kendall has accorded this writer support by insisting that the saturation index is not a direct measure of the corrosivity of water. It was proposed, and I think clearly explained in the writer's paper, that this index is "*a measure of directional tendency and driving force*" whether toward corrosion or scaling effect and was recommended as an analytical guide to be used in corrective treatment.

This index, as its name implies, was not intended to answer the question as to whether a water is or is not corrosive. The answer to this question is much simpler and for normal water containing dissolved air can be answered usually in the affirmative. In this connection it might be mentioned that the often recommended test of suspending a nail or wire in a bottle of water to ascertain its corrosivity is practically useless, because under such conditions a normal water of zero or even slightly positive index is bound to corrode. This, of course, is partly due to the lack of precipitating or buffering capacity of the fixed volume of water which permits the index to change as corrosive action proceeds.

Mr. Kendall's Fig. 1 might be taken to indicate that so far as pitting or the life of hot-water piping is concerned, the advantages of corrective treatment, if any, would be limited in many cases to comparatively new pipe. Also, it is interesting to note that during the first 4 yr. of service two supplies of widely divergent mineral character (Los Angeles and New York) show practically the same rate of pitting. These are important disclosures, and if additional data with respect to the life of hot water storage tanks and the occurrence of red-water troubles are found to conform therewith, the usefulness of the carbonate saturation theory might well be questioned.

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[2] For description of method see paper by H. E. Searle and F. L. LaQue, "Corrosion Testing Methods," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 249 (1935).

[3] Professor of Sanitary Engineering, University of California, Berkeley, Calif.

In the matter of direct relationship between specific conductivity of water and its corrosivity, the evidence is not as convincing. In Fig. 3 are plotted average results of four tests for the New York supply and one test for the Los Angeles supply. If instead there had been plotted the five tests for the New York supply and the three tests for the Los Angeles supply—all made within the second, third, and fourth years of service—it would seem that the two extremes in conductivity approximate the same corrosivity. It is generally agreed that in practice distilled and sea waters are both highly corrosive, yet these represent extremes in specific conductivity. In the writer's opinion, it would be more likely that the current strength in corrosion cells is determined by the physical and chemical conditions at the anode and cathode surfaces rather than by water conductivity.

MESSRS. J. C. WARNER[4] and D. S. MCKINNEY[4] (*presented in written form*).—We were especially interested in the linear relation between corrosivity and conductivity reported by the author. In the absence of analyses of these supplies, one can only speculate on the reasons for the observed relation. Either the waters may contain ions contributing to conductivity in approximately constant rates (that is, all samples may be considered dilutions of the same water) or the ratio of the concentration of some corroding agent to conductivity may be constant for the various supplies. We would like to ask Mr. Kendall whether any such relationship has been noticed.

MR. F. N. SPELLER.[5]—Mr. Kendall's data are based on depth of penetration while Mr. LaQue's are based on loss in weight. Although most of Mr. LaQue's specimens were penetrated, the extension of the curve confirms Mr. Kendall's curve. The pitting factor, which as you know is the maximum depth of pitting divided by the average rate of corrosion determined from loss in weight, has been found to be of considerable importance in soil corrosion. We should like to ask whether the author has any information as to what the pitting factors would be in these tests. They would probably vary considerably. It is desirable in corrosion tests to determine both the loss in weight and the depth of pitting. It is quite possible that pitting may be associated with certain types of waters and uniform corrosion with others.

MR. R. M. PALMER.[6]—In the case of the New York City supply and the Baltimore supply, the New York City supply is an untreated water, while the Baltimore supply is treated with lime. As I understand Mr. Kendall's curves for the hot water supply, it is fair to assume that the treatment used in Baltimore is of no value. Is that correct—for hot water?

MR. V. V. KENDALL[7] (*Author's closure*).—The data offered by Mr. LaQue are most interesting in that it also indicates that most of the pitting occurs in the initial exposure. Replotting the data to show total depth of pitting *versus* period of exposure does not show the decrease in the rate of corrosion as clearly as the rate chart given in the text. However, the total depth of pitting *versus* period of exposure is shown in the accompanying Fig. 2.

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[4] Professor of Chemistry and Assistant Professor of Chemistry, respectively, Carnegie Institute of Technology, Pittsburgh, Pa.

[5] Consulting Metallurgical Engineer, Pittsburgh, Pa.

[6] President, Ferro-Nil Corp., New York, N. Y.

[7] Corrosion Engineer, National Tube Co. (Subsidiary of U. S. Steel Corp.), Pittsburgh, Pa.

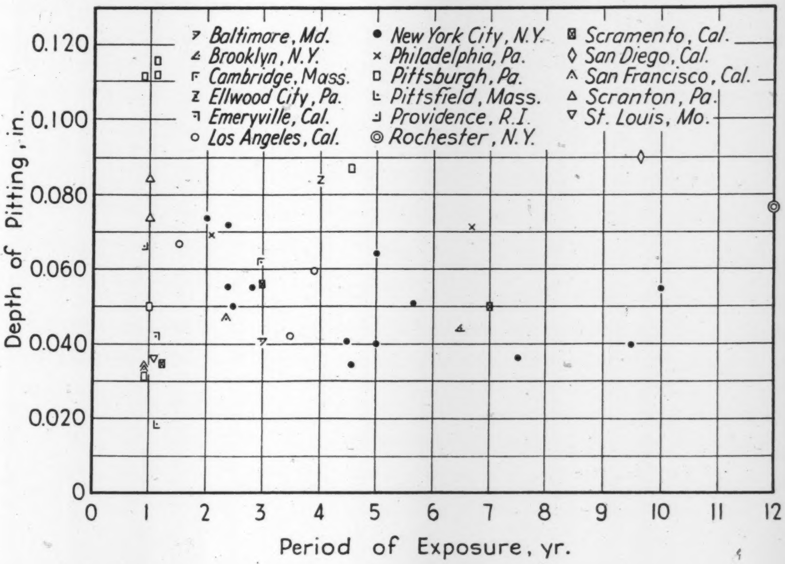

FIG. 2.—Data from 42 Service Tests in Hot Water Lines in 17 Cities.

In regard to the question of the pitting factor raised by Mr. Speller, in practical service tests such as these, it is impossible to obtain a loss in weight figure sufficiently accurate to be of value. The pitting factor would undoubtedly vary considerably for different kinds of waters. It would also probably vary in the same water depending on the period of exposure due to the decrease in the rate of pitting. The fact that most of Mr. LaQue's 0.05 in. thick specimens were perforated when removed emphasizes the point that corrosion test specimens should be sufficiently thick to provide for a high initial rate of pitting, in which case both loss of weight and pitting can be measured for his type of specimens.

Some possible misinterpretations of these data have been brought out in the discussion. The fact that there is a marked decrease in the rate of pitting to a possibly constant very low rate does not mean that pipe will last the same length of time in all waters. Neither does it mean that after a period of three or four years corrosion ceases. In the case of the very corrosive waters, corrosion will probably still continue but will be very considerably slowed down. In the case of the slightly corrosive waters, corrosion may actually cease. That action would depend upon the character of the initial rust coating. If it is porous, in the form of tubercles such as is generally found in very corrosive waters, corrosion will continue. If it is dense, adherent, and, in the case of waters that have received corrective treatment, backed up with some calcium carbonate, corrosion will cease entirely. While the curve given is the average of a considerable amount of data, there is still not enough data on each water for different periods of exposure. If such data were available we would probably have a curve for each water, similar to the average curve but placed at different distances from the base line. This would answer the question raised by Mr. Palmer. New York City water, untreated for corrosion prevention, gives a certain curve. Baltimore, with corrective treatment, would give a curve much lower than that for New York City. Only one point, at three years which is lower than the average curve, is given for Baltimore and, considering the experimental variations, the probabilities are that other points would place the curve for Baltimore much lower.

The value of corrective treatment is not limited to new pipe as suggested by Mr. Langelier. Corrective treatment in the case of pipe on which the rust coating has already formed would stabilize this coating and probably stop the continuation of corrosion. "Red water" or water in which iron or rust is present shows that disintegration of the rust coating with consequent further attack of the underlying surface is in progress. Corrective treatment stops this action, stabilizes the rust coating and, while it may not entirely stop corrosion, certainly slows it down. The value of the carbonate saturation theory is not questioned. In our experience, we have seen too many cases of natural thin coatings with no corrosion beneath them to doubt their value. It is the region between no natural coating and a definite coating, that is, at and near the carbonate saturation point, that we are investigating.

The conclusion that we can draw from these data is that the rate of corrosion decreases very considerably with time—as a matter of fact, much more rapidly than we would anticipate. An interesting case substantiating this was recently brought to our attention. Examination of a feedwater line to a newly installed industrial boiler led to a survey of its condition. The line had been in about 17 months, 5 months of which was actual operating time, and was carrying hot softened water. Corrosion was severe and it was calculated on the basis of the pitting measurements made that the line would not last more than 5 yr. longer. After 10 yr. it is still in service. No changes in operating conditions that would affect the corrosivity of the water have been made.

In reply to the question by Messrs. Warner and McKinney, we did not find any ion or ratio of ions that showed any direct correlation either to corrosivity or conductivity. This question is still under investigation. The correlation between corrosivity and electrical conductivity is offered simply as a subject worthy of further consideration and research. We believe that more accurate data will show a more definite correlation but whether that correlation will be sufficient to aid us in forecasting the corrosivity of a water depends on more accurate and more comprehensive data than we have at present.

MEASURING THE SCALE-FORMING AND CORROSIVE TENDENCIES OF WATER BY SHORT-TIME TESTS

By Everett P. Partridge[1] and G. B. Hatch[1]

Synopsis

It is recommended that the utility of a water for industrial purposes be judged, not on the basis of chemical analysis alone, but only after analysis has been supplemented by actual tests of the stability and corrosivity of the water. This is considered necessary because very low concentrations of some substances may greatly reduce the tendency of a water to precipitate a solid phase or to attack metal confining surfaces.

Short-time tests are classified as batch or continuous-flow, and the applicability of each type to the determination of stability and corrosivity is discussed. Emphasis is placed upon proper understanding of the essential factors in any test; this is illustrated specifically by the necessity of continuous-flow tests in measuring the ability of 2 ppm. of sodium hexametaphosphate to prevent the deposition of calcium carbonate in a distribution system, and to minimize the corrosion of steel.

It is pointed out that the rate of supply of an inhibitor, which is a function both of the rate of flow and of the concentration, is the factor to be considered rather than the concentration alone, when the inhibitor functions by adsorption upon a solid surface.

One who has to judge the utility of many different water supplies for industrial use generally develops a scheme of classification, which, though it may be arbitrary and approximate and may differ in detail from the schemes of other equally competent persons, reflects in a rough manner the experience of all. Few experts will agree exactly where to place the boundaries between different types of water; on the other hand, few would question that the water designated A in Table I would deposit a scale of calcium carbonate in a distillery condenser, or that water B would induce rapid corrosion of iron pipe and cause complaints from "red water."

Table I.—Typical Analyses of Scale-forming and Corrosive Water Supplies.

	Water A, Scale-forming Deep-Well Water, ppm.	Water B, Corrosive Surface Water, ppm.
Carbonate (CO_3)	0	0
Bicarbonate (HCO_3)	415	10
Sulfate (SO_4)	194	43
Chloride (Cl)	15	10
Silica (SiO_2)	17	....
Iron (Fe)	0.05	0.2
Calcium (Ca)	116	16
Magnesium (Mg)	58	2
Soap Hardness ($CaCO_3$)	530	48
Oxygen (O_2)	....	12.7
pH	....	6.0

Perhaps the most interesting fact about these diagnoses is that the introduction of 2 ppm. of the molecularly dehydrated phosphate usually known as sodium hexametaphosphate into each of the waters confounds good judgment by preventing scale deposition from water A and minimizing corrosion and virtually eliminating iron staining of plumbing fixtures by water B. This threshold treatment with sodium hexametaphosphate, based upon the discovery of Rosenstein[2] and developed by the authors and their associates, has been described in detail elsewhere.[3, 4, 5, 6] The purpose of the present paper is not to review the technical achievements of threshold treatment, but rather to offer the argument that, if 2 ppm. of any substance can profoundly alter the behavior of a water, any attempt to classify waters with respect to their use for various industrial purposes should be based not only upon inspection of routine chemical analyses but also upon actual tests of behavior under conditions closely simulating those in practice. Such tests may be expected to reveal differences in behavior due to substances other than hexametaphosphate present in small amounts—for example, organic matter resulting from the decay of vegetation or from the disposal of industrial wastes.

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[1] Director of Research, and Research Chemist, respectively, Hall Laboratories, Inc., Pittsburgh, Pa.

CLASSIFICATION OF TESTS

Tests of the behavior of water may be classified in several different ways. The two properties of greatest interest to the engineer are the tendency to deposit a solid phase when heated or chemically treated, and the tendency to corrode metal confining surfaces. Attempts may be made to evaluate these tendencies by either batch or continuous-flow tests. Such tests may extend over long periods of time or may be accelerated in the attempt to learn in a few hours or a few days what is likely to happen during years of normal service. Finally, in the case of corrosion, attention may be directed either to the rate of total loss of metal or to the rate at which the metal is perforated by a pit.

Still other methods of classification are possible, but the preceding will serve as an adequate basis for the present discussion. This will be limited to consideration of relatively rapid methods of testing the stability and corrosivity of industrial waters and the necessary precautions in the choice of a method.

RAPID TESTS OF STABILITY

Broadly speaking, a water may be defined as stable in any industrial operation if no solid phase is precipitated as a result of whatever thermal or chemical changes are produced in the operation. The solid phase most frequently encountered is calcium carbonate, but a water may also be considered unstable if it tends to precipitate hydrated iron oxide or calcium sulfate or even such less common substances as sulfur under the conditions of use.

In a plant already in operation, it is not difficult to determine by chemical analysis of water samples taken at appropriate points in the system whether a suspected solid phase is or is not depositing. Less simple is the problem of predicting before hand what a given water supply will do in a given operation. Parenthetically, it may be asked why, all too often, design engineers regard water as that abstract entity devoid of dissolved gases and solids, represented by the formula H_2O, rather than as the universal solvent which is likely to be either scale-forming or corrosive, if not both.

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[2] L. Rosenstein, U. S. Patent Reissue 20,754.

[3] G. B. Hatch and Owen Rice, "Surface-Active Properties of Hexametaphosphate," *Industrial and Engineering Chemistry*, Vol. 31, pp. 51-57 (1939).

[4] Owen Rice and E. P. Partridge, "Threshold Treatment," *Industrial and Engineering Chemistry*, Vol. 31, pp. 58-63 (1939).

[5] John O. Olson, "Station K Wins Battle Against Condenser Scale," *Power*, Vol. 84, pp. 84-85 (1940).

[6] G. B. Hatch and Owen Rice, "Corrosion Control with Threshold Treatment," *Industrial and Engineering Chemistry*, Vol. 32 (1940).

Batch Tests:

Probably the best-known of the stability tests is the "marble" test used to determine whether a water is saturated with respect to calcium carbonate.[7] When finely-divided calcium carbonate is agitated in a closed container with a sample of water which is not saturated, it will dissolve until the solution attains saturation. If, however, the water is initially supersaturated, as is the effluent of the typical cold-process lime or lime-soda softener, the solid particles added serve as nuclei to bring the water down to saturation. In either case, the change in the alkalinity of the water, as determined by titration to the methyl orange endpoint, is a measure of the degree to which the water initially deviated from a condition of equilibrium.

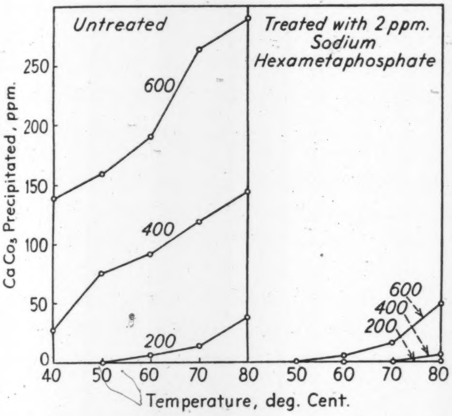

FIG. 1.—Inhibiting Effect of 2 ppm. of Sodium Hexametaphosphate on Precipitation of Calcium Carbonate.

Synthetic waters prepared by mixing equivalent amounts of calcium chloride and sodium bicarbonate to produce the indicated initial hardness as $CaCO_3$ were heated for 1 hr. at the respective temperatures.

This same simple titration of alkalinity may also be used to measure the extent to which calcium carbonate precipitates when a sample of water is heated or is aerated. In so far as conditions in a plant operation may be simulated in a batch test in the laboratory, it is thus possible to gage the relative stability of alternative sources of water or the relative efficacy of different treatments when only one source is available.

This general method was used by Hatch and Rice[3] in testing the effect of threshold treatment on synthetic waters. Figure 1, derived from their work, shows the amount of calcium carbonate precipitated on heating for 1 hr. at various temperatures mixtures of calcium chloride and sodium bicarbonate equivalent to the nominal concentrations of calcium carbonate indicated on the curves. The same type of data may be secured rapidly for a water supply, using any desired combination of time and temperature.

A somewhat elaborate system of calculating the extent of undersaturation or supersaturation has been described by Langelier,[8] who has defined as the "calcium carbonate saturation index" the difference between the actual pH of a water and its pH computed for a condition of equilibrium with solid calcium carbonate. This is more generally referred to as the "Langelier index." While Langelier has developed equations for calculating the pH at saturation, a direct determination might be preferred.

Continuous-Flow Test:

The calcium carbonate stability test has recently been converted from a batch to a continuous-flow operation by

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[7] The origin of this test is given by J. Tillmans and O. Heublein, "Investigation of the Carbon Dioxide Which Attacks Calcium Carbonate in Natural Waters," *Gesundheits-Ingenieur*, Vol. 35, pp. 669–677 (1912) as C. Heyer, "Ursache und Beseitigung des Bleiangriffes durch Leitungswasser (Cause and Prevention of the Attack of Tap Water on Lead)," Verlag Paul Baumann, Dessau (1888).

[8] W. F. Langelier, "The Analytical Control of Anti-Corrosion Water Treatment," *Journal*, Am. Water Works Assn., Vol. 28, pp. 1500–1521 (1936).

Enslow,[9] who pointed out the convenience of passing the water under investigation slowly through a column packed with calcium carbonate and comparing the alkalinity or pH of the influent and effluent water. Satisfactory experience with the Enslow continuous stability indicator has been reported by Leveque,[10] who used the arrangement indicated in Fig. 2.

To the extent that it has caused the water flowing through it to approach equilibrium, every filter following a cold-process lime or lime-soda softener has been functioning as a potential stability indicator to measure the supersaturation of the water leaving the settling basin. The time of contact with the grains of sand incrusted with calcium carbonate is, however, usually insufficient to bring the water close to equilibrium. This same situation may be encountered in the laboratory with a continuous stability indicator unless sufficiently long contact with an adequately large exposed surface of calcium carbonate is insured by comparison batch tests.

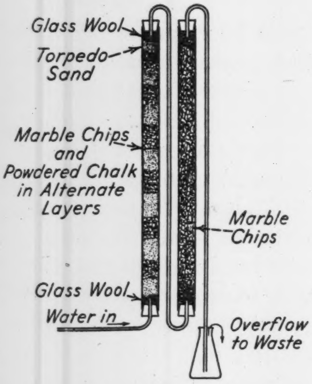

FIG. 2.—Diagram of Enslow Continuous Stability Indicator as Used by Leveque.

Either in the filter bed or in the laboratory test, the calcium carbonate surface may gradually become inactive as a result of the progressive adsorption upon it of small amounts of substances present in the water, the resultant film, even though invisible to the eye, effectively isolating the surface from actual contact with the water. An illustration of the change in behavior produced by as little as 0.5 ppm. of sodium hexametaphosphate, presented originally by Rice and Hatch,[11] is given in Fig. 3. The unstable water, supersaturated with calcium carbonate, consistently precipitated about 30 ppm. of this substance as it flowed continuously through a column containing sized particles of incrusted filter sand. Introduction of sodium hexametaphosphate, however, caused a steady decrease in precipitation, until the water was completely stabilized. With progressively higher concentration of sodium hexametaphosphate, the time required to stabilize the water progressively decreased. The inverse proportionality between concentration and time suggests that a certain minimum amount of the metaphosphate had to be supplied to the surface to coat it so that no further deposition could take place upon it.

Choice of a Test Method:

Judgment in setting up a test is just as important as accuracy in analysis. Particularly in testing the stabilizing effect of substances which inhibit precipitation by an adsorptive process, it is essential that the relation between the extent of the surface to be isolated and the supply of the inhibitor to this surface be appreciated. For example, if the use of sodium hexametaphosphate were being considered to stabilize the effluent of a cold-process lime softener as it passed through a distribution system already well coated with calcium carbonate, the batch "marble" test might seem appropriate. Fundamentally, however, this test would not be applicable; while the surface of calcium carbonate scale might be adequately represented by the powdered calcium carbonate added to 1 liter of the softened water in a bottle, 1 mg. of inhibitor supplied by this liter would by no means represent the cumulative effect of this same concentration supplied continuously to the water flowing through the distribution system. In fact, the precipitation in the batch test might well lead to rejection of the proposed treatment, when a properly chosen continuous-flow test, such as that which produced the data in Fig. 3, would have demonstrated its true effectiveness.

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[9] L. H. Enslow, "The Continuous Stability Indicator," *Water Works and Sewerage*, Vol. 86, pp. 107–108 (1939); Vol. 87, Reference and Data Section, p. 102 (1940).

[10] R. J. Leveque, "The Enslow Continuous Stability Indicator," *Water Works and Sewerage*, Vol. 86, pp. 450–451 (1939).

[11] Owen Rice and G. B. Hatch, "Threshold Treatment of Municipal Water Supplies," *Journal*, Am. Water Works Assn., Vol. 31, pp. 1171–1185 (1939).

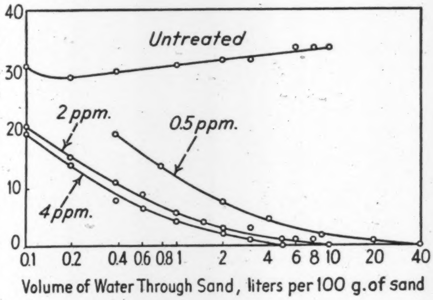

FIG. 3.—Inhibition of Precipitation of Calcium Carbonate on Incrusted Filter Sand by Sodium Hexametaphosphate in Continuous-Flow Test.

The effect of a continuous-flow test could, of course, be obtained by using the same portion of calcium carbonate repeatedly in successive batches of water treated with inhibitor. When this is done, progressively less precipitation occurs until complete stabilization is attained. Conversely, calcium carbonate used in the presence of inhibitor should not be re-employed in a test from which inhibitor is intended to be excluded.

It might be argued that the amount of inhibitor necessary to coat the surface of the calcium carbonate could be supplied as well by a relatively high concentration in a batch test as by a very low concentration in a continuous-flow test. Aside from the fact that sodium hexametaphosphate in higher concentrations actually has the ability to dissolve calcium carbonate in alkaline solution by forming a soluble complex with calcium ion, such a method of approach fails to reveal the all-important minimum concentration that would be necessary in practical use.

Although many cases in which stability must be measured can only be simulated properly by continuous flow, the utility of batch heating tests must not be discounted. When solid calcium carbonate is not initially present, and is developed only as a result of heating the water, the batch test offers a most convenient means of measuring the extent to which various treatments inhibit precipitation. If a test of this sort is conducted in a container other than glass, care must be exercised, however, that inhibitor adsorbed on the container surface during one test does not carry over into a subsequent test ostensibly without inhibitor.

RAPID TESTS OF CORROSIVITY

Much corrosion testing is concerned with the comparative resistance of different materials to a particular environment. The effect upon a given material of modifying the environment is, however, the chief concern of the engineer charged with the maintenance of existing equipment or with the construction of new equipment in which restrictions on capital investment limit him to the use of steel. How to evaluate the relative corrosivity of various water supplies or the protective action of different chemical treatments is a matter of no small interest to him.

As in the case of tests of stability, tests of corrosivity will be classified in this paper as either batch or continuous flow. The latter term will be restricted to tests in which the water comes in contact but once with the test surface and then leaves the test system. All other tests, whether conducted by simply hanging a test strip in a beaker of water or by recirculating water through pipe, are considered as essentially batch operations, since they comprise continued or repeated contact with the test piece of the water initially introduced into the system.

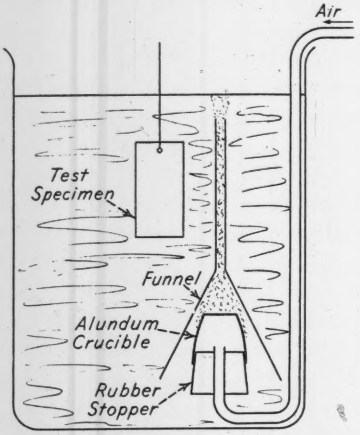

FIG. 4.—Use of Air in Batch Corrosion Tests to Promote Gentle Circulation and Maintain Saturation with Respect to Oxygen, as Suggested by Speller.

Batch Tests:

The simple and obvious device of immersing a strip of metal in a beaker of water has been employed widely, sometimes with little discrimination. Speller[12] has emphasized the importance of such factors as the volume of water, its oxygen concentration, and rate of movement past the test surface, and has suggested the use of compressed air, as indicated in Fig. 4, to keep the water saturated with oxygen and gently circulating. To simulate higher rates of flow, a cylindrical specimen may be rotated in the water at any practicable speed. In either case, the porous alundum crucible is desirable to produce small bubbles, and the inverted funnel to minimize direct contact of these with the test specimen.

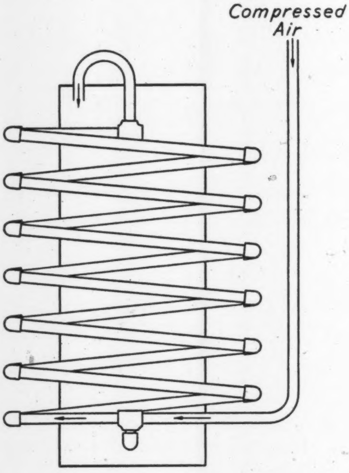

FIG. 5.—Diagram of Batch Recirculation Apparatus Using Pipe Bank for Testing Corrosivity of Water as Suggested by Speller.

Temperature should, of course, be controlled, or temperature variations should be known. Where it is impossible or inconvenient to control temperature closely, the effect of moderate variations can be cancelled out by comparing the results of a series of tests conducted simultaneously side by side. Beaker tests run at different times should not, however, be compared unless the temperature history of each sample is taken into consideration.

The beaker type of batch test can be carried out with simple equipment available in any laboratory, but reliance upon the loss in weight or depth of pitting of only one or two test pieces with only a few square inches of surface may lead the investigator astray. The increase in certainty with increase in the number of duplicate tests has been discussed from the statistical standpoint by Evans.[13]

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[12] F. N. Speller, "Corrosion: Causes and Prevention," Second Edition, Chapter VI, particularly pp. 225–230, McGraw-Hill Book Co., New York, N. Y. (1935).

A form of batch test more elaborate than the beaker test, but one approaching more closely, at least in a mechanical sense, the conditions in an industrial or municipal water distribution system, was described 24 yr. ago by Speller.[14] Figure 5 shows the essential details diagrammatically. Water is circulated by means of an air lift continuously through a bank of pipe lengths connected by return bends, discharging into the top of the open tank. The temperature of the water may be held at any desired level by means of a heater equipped with a thermostat.

Various adaptations of this sort of test might be made. For example, if the physical presence of air bubbles in the water passing through the test pipe were not desired, the water could be circulated by means of a pump, and aerated by means of a diffusion plate in the upper part of the tank, analogous to the porous alundum crucible in the beaker test illustrated in Fig. 4.

With this type of test, depth of pitting has been used as the criterion of corrosive attack, although loss in weight could be determined, if desired, by careful procedure in dismantling the pipe bank and removing corrosion products. The apparatus was well adapted to its original purpose of comparing the relative corrosion resistance of different materials, as, for example, wrought iron and steel, lengths of which could be placed alternately in the pipe bank. To test two different waters, or the same water subjected to different methods of treatment would, however, require either expensive duplication of equipment or an unduly extended time.

In both types of batch test just described, constancy of oxygen concentration is attained by keeping the water saturated at the test temperature. This at the same time tends to maintain the maximum rate of corrosion possible under the other experimental conditions.

TABLE II.—COMPOSITION OF RAW WATER AND COMPUTED COMPOSITION OF WATER SOFTENED BY ZEOLITE AND COLD INTERMITTENT LIME-SODA PROCESSES.

	Water A Raw		Water B Zeolite		Water C Cold Intermittent Lime-Soda	
	ppm.	epm.	ppm.	epm.	ppm.	epm.
Hydroxide (OH)	0	0.00	0	0.00	6	0.33
Carbonate (CO_3)	0	0.00	0	0.00	21	0.70
Bicarbonate (HCO_3).......	118	1.93	118	1.93	0	0.00
Sulfate (SO_4)...	42	0.88	42	0.88	42	0.88
Chloride (Cl)...	56	1.57	56	1.57	56	1.57
Silica (SiO_2)....	6–7	....	6–7	....	6–7	....
Calcium (Ca)...	47	2.33	2	0.10	6	0.30
Magnesium (Mg)........	2.3	0.19	0.5	0.05	1	0.08
Sodium (Na), calculated....	43	1.86	97	4.23	71	3.10

Aeration may, however, produce an undesirable change in composition of a water, thereby affecting its corrosive behavior. For example, if comparative tests were being run to determine the corrosivity of a water high in bicarbonate, such as the raw water A in Table II, with that of the same water after softening with zeolite, B, or with lime and soda, C, one would expect continued aeration to remove some carbon dioxide from both A and B, thereby increasing their pH values, while C would absorb carbon dioxide with a corresponding decrease in pH. Changes might also occur as the result of the deposition of calcium carbonate or the loss of corrosion inhibitor by adsorption. The steady state attained by each of these waters would, of course, depend upon the concentration of carbon dioxide or of other acid gases, such as sulfur dioxide, in the air; the composition and quite possibly the characteristics of the water actually tested might differ considerably from those of the water supposedly under test, and actually to be used in plant operation. Even where a water is unlikely to suffer any material change as a result of aeration it would, therefore, seem wise to check its composition at intervals during a test.

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[13] U. R. Evans, "Metallic Corrosion, Passivity and Protection," pp. 665–667, Edward Arnold and Co., London (1937).

[14] F. N. Speller, "Method for Testing the Durability of Pipe Under Corrosion," *Proceedings*, Am. Soc. Testing Mats., Vol. XVI, Part II, p. 343 (1916).

Continuous-Flow Tests:

Aside from the question of time, the objections to the recirculation of a limited volume of a water over a test specimen or through a test pipe could, of course, be met by setting up a once-through system in which the water, after contact with the test surface, was used for ordinary purposes or run to waste. In a once-through system there could be no uncertainty as to whether the water during a test differed in corrosivity from the water supplied. Moreover, if the water were derived from a source which was variable with respect to composition and corrosivity, once-through tests automatically would integrate the effect during the test period.

This type of test, using drop in oxygen concentration as a measure of corrosion, has been described by Speller and Kendall.[15] Using test sections made from 200 ft. of ¼-, ½-, and ¾-in. steel pipe, they found that the rate of corrosion increased with both temperature and water velocity.

Whitman and Russell[16] also employed the oxygen-drop method but used as the test surface steel plates arranged as baffles in a cypress box through which Cambridge tap water flowed continuously.

To minimize both the initial cost of equipment and the water requirements, Hatch and Rice[6] substituted steel wool as the test medium in a once-through system. Their relatively simple apparatus is shown in Fig. 6. The rate of water supply to each unit is governed by an ordinary screw clamp on a rubber tube connected to the constant-head tank. As the water is delivered into the head tube it receives any desired chemical treatment from a constant-head dispenser through a siphon containing a capillary section to limit the rate of flow of the treating solution to approximately 0.5 to 1 per cent of the rate of flow of the water under test. The treated water passes down the head tube, through the air trap to remove entrained bubbles, then upward through 20 g. of No. 3 steel wool, packed into 6 in. of a glass tube with a diameter of 1¼ in., and finally overflows from the sample bottle. An-

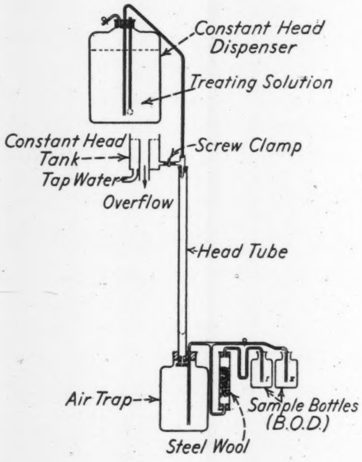

FIG. 6.—Continuous-Flow Apparatus Using Steel Wool for Testing the Corrosivity of Water by the Oxygen-Drop Method.

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[15] F. N. Speller and V. V. Kendall, "A New Method of Measuring Corrosion in Water," *Industrial and Engineering Chemistry*, Vol. 15, pp. 134–139 (1923).

[16] W. G. Whitman and R. P. Russell, "The Natural Water Corrosion of Steel in Contact with Copper," *Industrial and Engineering Chemistry*, Vol. 16, pp. 276–279 (1924).

other bottle is connected to allow sampling just ahead of the steel wool.

The purpose of the head tube is to indicate the increase in resistance at constant rate of flow as corrosion products tend to plug the interstices of the steel wool. The measurement of head and the visual appearance of the steel wool during a test supplement the measurement of the drop in oxygen concentration by indicating to some extent how, as well as how much, corrosion is occurring. The amount of iron picked up by the water on passing through the steel wool may be determined, if desired, but the drop in oxygen concentration is considered to be the criterion of the rate of corrosion.

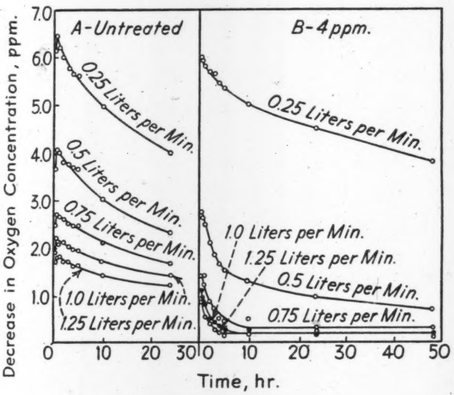

FIG. 7.—Decrease in Oxygen Concentration at Various Rates of Flow in the Absence and Presence of Sodium Hexametaphosphate.

Pittsburgh tap water at room temperature was passed continuously through 20 g. of No. 3 steel wool.

By substituting for the steel wool in Fig. 6 a 6-in. strip of thin sheet rolled up into a loose bundle with reasonably constant spacing between surfaces, the type of attack, whether general or localized, and the number and depth of pits formed during a short-time test may be observed. While the general method is suitable over a wide range of oxygen concentrations, some modification of the actual apparatus would be necessary if the water to be tested contained more or much less oxygen than that corresponding to saturation at atmospheric pressure.

Given the problem of trying to minimize the corrosion of a specified water at some particular temperature, the experimental variables are the rate of flow and the chemical treatment, the oxygen concentration being that naturally present in the water. Once a rate of flow has been chosen which will produce a convenient drop in oxygen content of perhaps 2 ppm. at the start of a test, the possibility of increasing the pH or of feeding various inhibitors may be investigated in tests run simultaneously to cancel out variations in the natural corrosivity of the water.

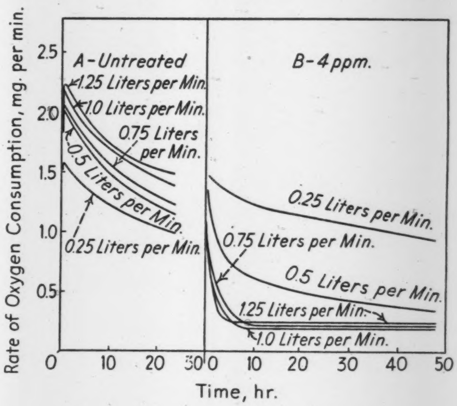

FIG. 8.—Derived Curves Showing Effect of Sodium Hexametaphosphate on Rate of Corrosion as Measured by Rate of Oxygen Consumption.

These curves are derived from those of Fig. 7.

A few illustrative groups of data obtained with this method of testing are shown in Figs. 7 to 10. The results are plotted against time in two different ways. Figures 7, 9, and 10 are expressed in terms of the measured decrease in oxygen concentration of Pittsburgh tap water passing through 20 g. of steel wool. To produce Fig. 8 this decrease has been multiplied by the rate of flow to give the rate of removal of oxygen, which is a measure of the rate of corrosion.

Figure 8 illustrates the relative importance of the rate of supply of the corrosion accelerator, oxygen, and of the corrosion inhibitor, sodium hexametaphosphate. In the left-hand group of curves, the rate of corrosion is progressively higher for increasing rates of flow; from the right-hand group of curves it is evident that increasing the rate of supply of the inhibitor by increasing the rate of flow far more than counterbalances the corresponding increase in the rate at which oxygen is brought to the metal surface.

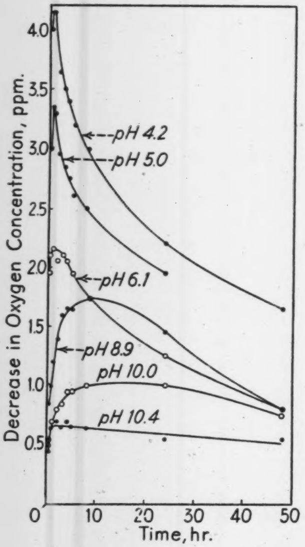

FIG. 9.—Effect of pH upon the Corrosive Behavior of Untreated Pittsburgh Tap Water.

Water was passed continuously through 120 g. of No. 3 steel wool.

Increase in pH has commonly been employed in the past to reduce corrosion of steel by water. Even in waters where there could be little expectation of forming a coating of calcium carbonate by this treatment, some protection has been obtained, presumably by the development of a more dense and more tightly bonded layer of corrosion products on the steel. For example, the addition of increasing amounts of caustic soda to Pittsburgh tap water was found by Speller and Texter[17] to decrease the rate of oxygen removal from the water at 150 F. during continuous-flow experiments in which a test section of 50 ft. of ½-in. pipe was employed. Continuous-flow studies by Hatch and Rice[6] on Pittsburgh tap water adjusted to various pH values yielded the series of curves in Fig. 9 when no sodium hexametaphosphate was added and the series in Fig. 10 when 2 ppm. of this inhibitor were maintained in the water. The relative position of the curves in Fig. 9 does indicate a general progressive decrease in rate of corrosion with increase in pH, but even at the maximum pH value of 10.4, the corrosion levels off at a higher rate than

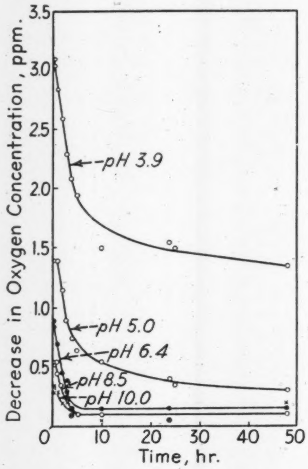

FIG. 10.—Effect of pH upon the Corrosive Behavior of Pittsburgh Tap Water Treated with 2 ppm. of Sodium Hexametaphosphate.

Water to which 2 ppm. of sodium hexametaphosphate had been added was passed continuously through 20 g. of No. 3 steel wool.

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[17] F. N. Speller and C. R. Texter, "Effect of Alkaline Solutions on the Corrosion of Steel Immersed in Water," *Industrial and Engineering Chemistry*, Vol. 16, pp. 393–397 (1924).

at a pH of 5.0 in Fig. 10 in the presence of what might be termed by the uninitiated an insignificant trace of metaphosphate. The effect of 2 ppm. of this inhibitor is so much greater than that of increase in pH that the curves for pH values of 6.4, 8.5, and 10 in Fig. 10 do not deviate from one another by more than the experimental uncertainty, except during the first few hours when the protective film is being established.

Choice of a Test Method:

Because water, in general, is kept moving in an industrial or municipal water system, the authors believe that continuous-flow tests will, to a progressively increasing extent, replace batch tests in engineering corrosion studies. As noted previously in connection with the testing of stability, wherever the effect of an inhibitor such as sodium metaphosphate upon an existing surface is to be investigated, only a continuous-flow test will properly indicate the degree of protection attainable and the minimum concentration required.

One essential factor in the testing of an inhibitor that exerts a cumulative action by adsorption to form an integral part of the protective film is the maintenance of a reasonable relation between the area of the test surface and the rate of supply of the inhibitor to this surface by the flowing water. Theoretically, the ratio of the mass velocity of the water in pounds per minute to the area of surface exposed in square feet should be approximately the same in a test as in the plant operation under consideration; actually, if steel wool is used as the test medium a considerably lower ratio may be used because the contact of the water with the surface is much more complete in passing through a 20-g. plug of No. 3 steel wool than through a length of pipe with the equivalent internal surface of approximately 1.8 sq. ft. Hatch and Rice found a flow of from 0.6 to 1.2 lb. per min. per sq. ft. of surface suitable in their tests.[6]

APPLICATION AND INTERPRETATION OF SHORT-TIME TESTS

To the engineer who demands a definite figure in inches per year for the rate of scale formation or the rate of penetration if a particular water is employed in a certain operation, the short-time tests described in the preceding sections may have little to offer. The importance of these tests lies not in their ability to yield absolute values but, instead, relative values and quick values. Considered together with cost data, relative values for the effectiveness of various treatments in preventing scale and corrosion should certainly suffice for a decision concerning a problem in an existing operation. The question of whether any treatment at all will be required in an operation not yet off the drafting board may also be answered in many cases by comparing, in simultaneous short-time tests, the relative behavior of the water supply in question with that of other supplies for which prior operating experience is available.

In conclusion, it may be reiterated that short-time tests, when properly designed to simulate the fundamental factors in an operating problem, will yield more certain information concerning the relative stability and corrosivity of different waters or of the same water subjected to different treatments, than can be derived from chemical analyses of the waters alone. Short-time tests, to be of practical value, must, however, be supervised and interpreted by someone with a clear understanding of the chemical processes and engineering principles involved.

DISCUSSION

MESSERS. J. C. WARNER[1] **AND D. S. MCKINNEY**[1] (*presented in written form*).—A casual reading of this paper gave us the impression that water analysis is of little value in predicting the behavior of a supply in actual service, especially with regard to rates of corrosion and scale deposition. We believe, however, that it was the authors' intention to point out that our knowledge of inhibitors and accelerators is so inadequate that we do not know what determinations to make in order properly to judge the quality of the water from its analysis. The short-time tests described in this paper are valuable, not only in evaluating the effects of known inhibitors as demonstrated by the authors, but in detecting natural inhibitors or accelerators in water supplies. This may be done by comparing the behavior of a synthetic water, made from pure materials, with the naturally occurring supply of similar composition. Appreciable differences in the behavior of the natural and synthetic waters would indicate the presence of inhibitors or accelerators. The natural supply should then be subjected to a more searching analysis than is customary. The materials thus discovered would be added, one by one, to the synthetic water and subjected to the short-time test. Such a program, although tedious, would eventually lead to sufficient knowledge of inhibitors and accelerators that accurate predictions could be made from analyses including these important substances.

The authors' discussion of the relative merits of batch and continuous tests is a valuable contribution to the literature of water testing.

MR. F. N. SPELLER.[2]—In his paper Mr. Partridge has frequently referred to the inhibiting effect of hexametaphosphate of soda, to illustrate what may be learned from short-time tests. However, I should like to ask him whether this inhibitor will operate favorably with moderately high chlorides in the water. The impression may get out that this inhibitor has a more general application than the data from his tests seem to warrant. I think a word as to the influence of the metaphosphate in waters of different compositions would be in order in the discussion.

MESSRS. EVERETT P. PARTRIDGE[3] **AND G. B. HATCH**[3] (*Authors' closure by letter*).—No objection should be raised to the suggestion of Messrs. Warner and McKinney; systematic investigation over a sufficiently long period of time could scarcely fail to reveal new information as to why apparently similar waters sometimes behave in a dissimilar manner. More than twenty years of scientific investigation has, however, not yet produced a reliable basis for predicting corrosion behavior from chemical analysis alone. In the meantime, the engineer has to make things work—and right away! Until analytical procedures can be used to predict functional behavior, direct functional tests will rightfully seem more convincing.

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[1] Professor of Chemistry, and Assistant Professor of Chemistry, respectively, Carnegie Institute of Technology, Pittsburgh, Pa.

[2] Consulting Metallurgical Engineer, Pittsburgh, Pa.

[3] Director of Research, and Research Chemist, respectively, Hall Laboratories, Inc., Pittsburgh, Pa.

Mr. Speller has asked specifically concerning the effect of chloride upon corrosion control by sodium hexametaphosphate. Some corrosion was originally expected when scale was eliminated in one condensing system with from 17,000 to 20,000 ppm. of chloride in the circulating water,[4] but this has not materialized. Similar encouraging results have been obtained in the cooling-water system of an oil refinery where the chloride concentration of one sample was 28,500 ppm. So much is still unknown, however, concerning inhibition of corrosion by means of threshold treatment that no general statement can be made.

The authors are as anxious as Mr. Speller that sodium hexametaphosphate should not be regarded as a cure-all. The laboratory studies from which the data in this paper were taken are being continued to cover a variety of conditions. In the meantime, the functional test with steel wool is recommended to the engineer anxious to know what will happen with his particular water.

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[4] J. O. Olson, "Station K Wins Battle Against Condenser Scale," *Power*, Vol. 84, pp. 84-85 (1940).

A METHOD OF MEASURING CORROSIVENESS

By J. H. Walker[1]

Synopsis

This paper describes and discusses a method of measuring corrosiveness which was developed by the Corrosion Committee of the National District Heating Association for the study of the corrosion problem in steam heating systems. The device used consists of three helical coils of bessemer steel wire mounted on a frame which can be inserted in the pipes of a heating system.

Although designed primarily for field measurements, the tester proved to be well adapted to a semi-laboratory study of the corrosiveness of the condensate formed from the steam supplied by the four district heating plants of the Detroit Edison Co., and the results of that study are given as illustrative of the use of the tester. The possibilities and limitations of this general method of testing the corrosiveness of a water are discussed.

The district heating industry has for many years been active in the study of corrosion in heating systems. The member companies of the National District Heating Association have conducted and sponsored some important research, and, while much work remains to be done, the problem has been greatly clarified and certain remedies have been found.[2,3,4]

Several years ago, when active studies were begun, the need was recognized for some method of measuring corrosion in heating systems during their normal operation. It was desired to study the effect on the rate of corrosion of such factors as the type of heating system, the method of operation, and the amounts of impurities in the steam and condensate. After much discussion and some preliminary experimentation, the tester shown in Fig. 1 was adopted. The method of use has been standardized,[5] and it has been employed throughout the country in hundreds of tests during the past eight years.

The tester consists of three helical coils made from bessemer steel wire of 0.05 in. diameter. The helical coils have an outside diameter of approximately $\frac{11}{16}$ in., and each has about 1.5 sq. in. of surface and weighs about 2.5 g The wire is furnished on special order and is made from an ingot that has been cropped between 35 and 40 per cent The specified composition is as follows:[6]

Carbon, per cent	0.08 to 0.10
Manganese, per cent	0.40 to 0.55
Phosphorus, max., per cent	0.10
Sulfur, per cent	0.07 to 0.08

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[1] Engineer Assistant to the General Manager, The Detroit Edison Co., Detroit, Mich.

[2] Leo F. Collins with Everette L. Henderson, "Corrosion in Steam Heating Systems," *Heating, Piping and Air Conditioning*, Vol. 11, September, 1939 to Vol. 12, May, 1940.

[3] R. E. Hall and A. R. Mumford, "Some Fundamental Considerations of Corrosion in Steam and Condensate Lines," *Transactions*, Am. Soc. Heating and Ventilating Engrs , Vol. 38, pp. 121-171 (1932).

[4] R. R. Seeber, F. A. Rohrman and G. E. Smedberg, "Corrosion Studies in Steam Heating Systems," *Heating, Piping and Air Conditioning*, Vol. 6, pp. 124-126 (1934); Vol. 8, pp. 103-111 (1936).

[5] The Measurement of Corrosion in Steam Heating Systems, a Progress Report of the Corrosion Committee, Nat. District Heating Assn. (1934). Not published, but copies available.

[6] Analysis by American Steel and Wire Co.

The wire is purchased in large lots to insure uniformity. It is pickled and annealed, and to avoid corrosion is kept packed in lime.

The method of installing the tester in the condensate pipe of a heating system is shown in Fig. 2. Such pipes usually run only partly full of condensate, and carry air and other gases. The tester is therefore installed in a loop so constructed that the tester is always submerged in the flow of condensate, while the gases pass through the upper pipe without restriction. In a pipe running full of condensate, this arrangement is unnecessary, and the tester is simply inserted into a pipe fitting or, if the pipe is large, into a threaded hole in the side of the pipe.

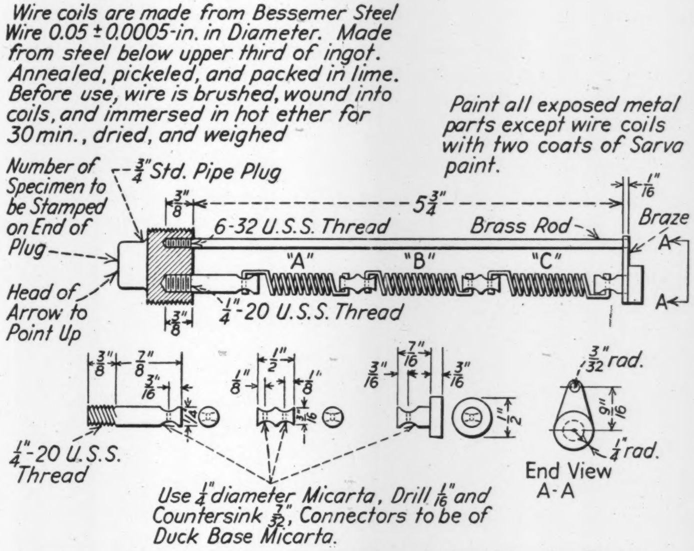
Wire coils are made from Bessemer Steel Wire 0.05 ± 0.0005-in. in Diameter. Made from steel below upper third of ingot. Annealed, pickeled, and packed in lime. Before use, wire is brushed, wound into coils, and immersed in hot ether for 30 min., dried, and weighed
Paint all exposed metal parts except wire coils with two coats of Sarva paint.
Number of Specimen to be Stamped on End of Plug
3/4" Std. Pipe Plug
3/8"
5 3/4"
1/16"
6-32 U.S.S. Thread
Brass Rod
Braze
A
"A"
"B"
"C"
Head of Arrow to Point Up
1/4"-20 U.S.S. Thread
3/8"
A
3/8"
7/8"
3/16"
1/4"-20 U.S.S. Thread
1/2"
1/8"
1/8"
3/16"
7/16"
3/16"
3/16"
1/2"
3/32" rad.
9/16"
1/4" rad.
End View A-A
Use 1/4" diameter Micarta, Drill 1/16" and Countersink 3/32", Connectors to be of Duck Base Micarta.

FIG. 1.—National District Heating Association Corrosion Tester.

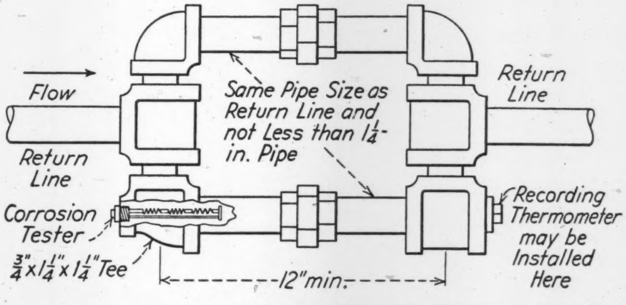
Flow
Return Line
Same Pipe Size as Return Line and not Less than 1 1/4-in. Pipe
Return Line
Corrosion Tester
3/4" x 1 1/4" x 1 1/4" Tee
12" min.
Recording Thermometer may be Installed Here

FIG. 2.—Method of Installing the N.D.H.A. Corrosion Tester in a Condensate Pipe.

The period of exposure found most satisfactory for measurements in steam heating systems is 28 days. This is long enough to give results which are accurately measurable, but not so long as to cause rupture of the wire.

At the end of the test period, the coils are withdrawn, the corrosion products removed by an acetic acid solution containing an inhibitor, and the loss in weight determined. The corrosion rate R is calculated from the loss in weight and, for convenience, is multiplied by 1000, giving values of $R \times 1000$ which usually fall between 1 and 100. Because the surface is cylindrical, it is necessary to take into account the fact that the area exposed to corrosion decreases during the test period. The corrosion rate R is therefore calculated by the following formula:[7]

$$R = \frac{D_1}{2T}\left(\frac{W_1 - W_2}{W_1 + \sqrt{W_1 W_2}}\right)$$

where:

R = corrosion rate in inches penetration per year,
D_1 = initial diameter in inches,
T = time of exposure in years,
W_1 = initial weight in pounds, and
W_2 = final weight in pounds.

Interpretation of Results:

The testers were made available to district heating companies and a large number of corrosion measurements were made and correlated. It was felt desirable to interpret the measured corrosion rates in terms of the practical seriousness of the corrosion effects in piping. Examination of the pipe surfaces in several cases, coupled with the histories of the progress of the corrosion, led to the following rough classification:

Values of $R \times 1000$ below 10 are termed "negligible."

Values of $R \times 1000$ between 10 and 20 are termed "mild."

Values of $R \times 1000$ above 20 are termed "active."

Any such classification is, of course, very approximate. In heating systems in most kinds of buildings, the seriousness of the corrosion effects corresponds roughly with the interpretation which would ordinarily be placed upon those words, and the terminology has been quite useful from a practical standpoint.

No attempt has been made to correlate the measured corrosion rate of the tester with any predictions as to the length of life of the piping. There are many obvious reasons why the corrosion rate of the tester would not be expected to correspond with the corrosion rate of the pipe. Nor has the tester been used to compare the corrosion rates of different materials. It has been regarded solely as a means of measuring the corrosiveness of the environment.

Mechanical Advantages:

This particular design of tester was adopted because of certain mechanical advantages, but there is nothing important about its exact form. It was desired primarily to have a device that could be readily inserted in the pipes of a heating system, and for that reason the specimens are mounted on a standard $\frac{3}{4}$-in. pipe plug and can be inserted in a 1-in. pipe. The helical shape of the specimens was chosen to reduce the length of the tester. The diameter and length of the wire were so selected as to make the loss-of-weight measurements significantly large in relation to the gross weight; and the diameter of the wire was also made sufficient to prevent complete destruction during the normal period of exposure. The basic design was inspired to some extent by a device proposed by Chapman.[8]

Agreement of Results from the Three Coils:

The three separate coils were used in order to eliminate possible errors of measurement and for confirmation of results. The agreement obtained between them is shown in Table I. There are appreciable differences in the corrosion rates of the three coils in several cases, but those differences are small compared to the differences produced by the different environments. It would be nearly as satisfactory to use a single coil, but the confirmation obtained from the three measurements is of value.

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[7] For derivation, see Appendix, p. 1352.

[8] Staff Article, *Power*, Vol. 74, August 11, 1931, p. 190.

AN EXAMPLE OF THE USE OF THE TESTER IN COMPARING CORROSIVENESS

The original purpose of the N.D.H.A. tester was to provide a large amount of data from which it was hoped to draw important conclusions as to the effect on corrosion of the design of the heating system and the method of operation. That work resulted in some valuable information, but the fact that there were certain unmeasured variables whose significance was not then appreciated has since thrown some question upon those conclusions. More recently, the tester has been used by the Detroit Edison Co. in some studies of a semi-laboratory nature which are presented here as a basis for a critical discussion of the tester.

TABLE I.—AGREEMENT AMONG THE THREE COILS OF THE TESTER WHEN PROPERLY INSTALLED.

Tester	Location of Tester	Corrosion Rate, Penetration in in. per yr., × 1000			
		Coil A	Coil B	Coil C	Average
No. 1	Building steam riser	6.8	6.0	8.6	7.1
No. 2	Drips from a low pressure distribution main	4.3	3.2	4.5	4.0
No. 3	Dry return line in a vacuum system	16.8	14.1	14.9	15.3
No. 4	Drips from a high pressure steam line	0.21	0.23	0.22	0.2
No. 5	Wet return line in a pressure system	19.1	22.5	22.2	21.3
No. 6	Return line from a unit heater	16.7	15.7	9.3[a]	16.2
No. 7	Dry return line in a pressure system	10.1	11.0	11.1	10.7
No. 8	Wet return line in a pressure system	22.0	19.5	21.3	20.9
No. 9	Measurement made to check tester No. 8	20.3	21.2	21.0	20.8
No. 10	Returns from a valet machine using high pressure steam	2.4	2.6	1.9	2.3
No. 11	Dry return line in a vacuum heating system	9.9	8.4	8.0	8.8
No. 12	Returns from a one-pipe pressure system	4.9	5.1	5.1	5.0
No. 13	Returns from a two-pipe atmospheric return system	9.1	9.0	8.2	8.8
No. 14[b]	Returns from a two-pipe atmospheric return system	14.6	14.5	6.3	6.3

[a] This coil was found to have been protected from the corrosive conditions and is not included in the average.

[b] This tester was located in one leg of a U-seal so that coils A and B were continually immersed, while coil C was immersed only when condensate was flowing. The results from coil C only are believed to be representative.

The condensate in heating systems may be corrosive if it contains, in solution, oxygen or carbon dioxide. The oxygen may be entrained in the steam or, if subatmospheric pressures are used, may enter the piping through leaks. Carbon dioxide is commonly present in steam generated from bicarbonate waters or water treated with carbonates.

The four district heating plants operated by the Detroit Edison Co. have different methods of feedwater treatment and use different proportions of make-up water. It appeared that if tests were made of the corrosiveness of the condensate formed from steam generated in each plant, the results should indicate the corrosiveness of the respective condensates and might throw some light on the question of the relative influence of carbon dioxide and oxygen.

The carbon dioxide and oxygen contents of the steam fluctuate, but representative average figures were as follows:

	O_2, ml. per liter	CO_2, ppm.
Beacon Street plant	0.0	16
Congress Street plant[a]	0.0	11
Congress Street plant[b]	0.0	3
Willis Avenue plant	0.43	25
Boulevard plant	0.64	2

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[a] Before change in feedwater treatment.

[b] After changing feedwater treatment to give more complete removal of CO_2.

It will be noted that the Beacon Street plant produced steam which was practically free of oxygen but contained appreciable amounts of carbon dioxide. The steam produced by the Congress Street plant formerly contained considerable carbon dioxide, but after changes were made in the feedwater treatment for that express purpose, the carbon dioxide was greatly reduced. The Willis Avenue plant steam was high in both oxygen and carbon dioxide, and the steam from the Boulevard plant was high in oxygen but low in carbon dioxide. The quantities of these gases were the natural result of the respective methods of feedwater treatment.

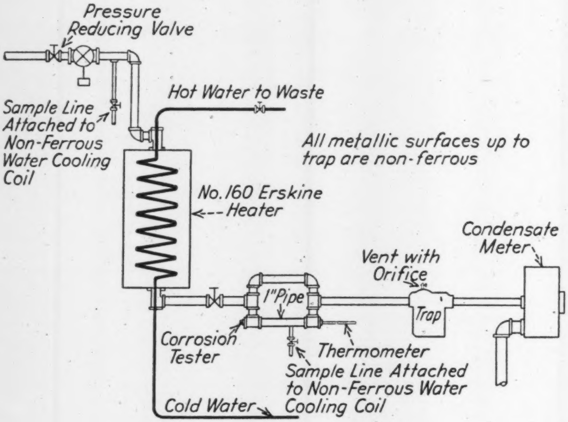
Pressure Reducing Valve
Hot Water to Waste
Sample Line Attached to Non-Ferrous Water Cooling Coil
All metallic surfaces up to trap are non-ferrous
No. 160 Erskine Heater
Condensate Meter
Vent with Orifice
1" Pipe
Trap
Corrosion Tester
Thermometer
Sample Line Attached to Non-Ferrous Water Cooling Coil
Cold Water

FIG. 3.—Corrosion Test Apparatus.

Test Apparatus:

It was necessary to make a separate test setup in each plant, and they were exactly alike in every important detail. The apparatus is shown diagrammatically in Fig. 3. The condenser was supplied with city water and with steam from the plant header. The condensate flowed through the pipe loop containing the corrosion tester and thence through a trap and meter. The pipe used for the loop was 1 in. nominal pipe size. All of the piping between the reducing valve and the meter, including the condenser, was of copper or brass, in order to avoid possible reduction of corrosiveness of the condensate by contact with iron. Tests of two to four weeks duration were run, during which periods several analyses of the condensate were made and the temperature and amount of condensate were measured. The rate of flow of condensate was adjusted to that desired for each test by regulating the flow of water to the condenser.

It soon became apparent that the corrosion rate increased greatly with increasing rate of flow of condensate over the tester. This fact had not been previously appreciated, and it is now evident that failure to take that variable into account in the earlier work with the tester in the heating systems of buildings renders those results of doubtful accuracy. Rate of flow is an important factor in the rate of corrosion of the N.D.H.A. tester.

Test Results:

The results of the entire series of tests are shown in Table II, but the correla-

TABLE II.—RESULTS OF CORROSION TESTS.

O_2 in condensate, ml. per liter[a]	CO_2 in condensate, ppm.	Condensate Flow Through Pipe Loop		Temperature of Condensate, Deg. Fahr.	Corrosion Rate, R, in. per yr., × 1000	$CO_2 \times V$
		lb. per hr.	Velocity, V, ft. per min.			
BEACON STREET PLANT						
0.0	3	47	2.19	...	3.2	6.38
...	....	100	4.67	...	7.8	......
...	....	236	11.0	...	17.7	......
...	....	260	12.1	...	18.0	......
...	....	318	14.8	...	21.9	......
0.0	3	231	10.8	224	18.4	32.4
0.0	3	142	6.66	224	11.5	20.0
0.0	4	620	29.0	224	26.4	116
0.0	8	611	28.6	224	38.2	229
0.0	4	427	19.9	224	22.6	79.6
0.0	5	90	4.20	220	8.3	21.0
0.0	5	60	2.80	220	3.6	14.0
0.0	20	100	4.67	...	36.5	93.4
0.0	14	69	3.22	...	17.9	45.0
0.0	8	655	30.6	...	37.9	245
0.0	12	934	43.7	223	61.0	522
0.0	16	545	25.5	223	48.6	408
0.0	16	515	24.1	223	39.2	385
CONGRESS STREET PLANT, BEFORE CO_2 CONTENT OF STEAM WAS REDUCED						
0.0	7	78	3.64	218	8.5	25.5
...	.	161	7.54	...	12.7	......
...	.	200	9.34	...	16.4	......
0.0	4	53	2.47	224	9.8	9.88
0.0	4	624	29.1	222	40.9	116
0.0	6	554	25.9	224	34.0	155
CONGRESS STREET PLANT, AFTER CO_2 CONTENT OF STEAM WAS REDUCED						
0.0	2	400	18.7	225	11.1	37.4
0.0	2	157	7.34	225	8.3	14.7
0.0	2	550	25.7	225	17.4	51.4
0.0	2	508	23.7	230	11.6	47.4
0.0	2	487	22.7	230	12.7	45.4
0.0	1	226	10.6	223	7.8	10.6
0.0	1	210	9.81	224	7.4	9.81
0.0	1	710	33.2	224	9.2	33.2
0.0	0.5	472	22.0	224	8.5	11.0
WILLIS AVENUE PLANT						
0.16	6	86	4.02	226	9.9	24.1
....	....	182	8.50	...	15.5	......
....	....	194	9.07	...	20.7	......
....	....	48	2.24	...	29.4	......
....	....	39	1.82	...	6.3	......
....	....	210	9.80	...	17.0	......
0.20	12	215	10.0	225	14.6	120
....	....	140	6.54	...	18.2	......
0.15	25	123	5.76	204	34.0	144
Trace	22	210	9.80	210	49.2	216
0.30	20	186	8.70	190	42.1	174
0.12	20	97	4.52	208	27.1	90.4
Trace	15	139	6.50	208	48.5	97.5
Trace	5	200	9.34	224	15.7	46.7
Trace	16	236	11.0	208	34.1	176
Trace	4	127	5.94	218	9.4	23.8
Trace	4	141	6.60	220	11.3	26.4
Trace	16	230	10.7	210	22.0	171
BOULEVARD PLANT						
0.31	1	79	3.69	225	6.0	3.69
....	....	50	2.34	...	4.1	......
....	....	149	6.95	...	6.8	......
....	....	270	12.6	...	7.3	......
0.30	1	156	7.28	224	6.5	7.28
0.26	1	58	2.71	224	3.3	2.71
0.03	1	217	10.1	221	4.4	10.1
0.20	2	75	3.50	209	8.2	7.00
0.20	1	108	5.08	219	4.1	5.08
Trace	1	108	5.08	219	3.3	5.08
0.24	2	150	7.01	219	8.8	14.02

[a] Winkler method.

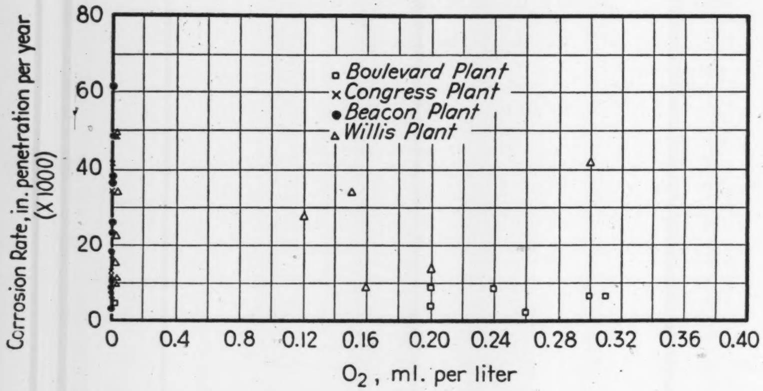

FIG. 4.—Relation of Corrosion Rate to Oxygen Concentration.

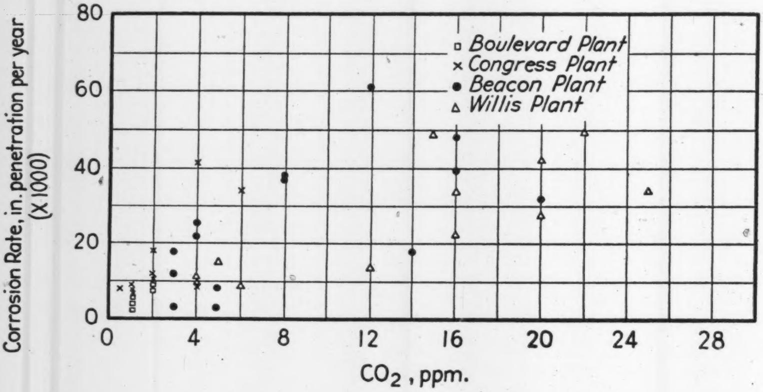

FIG. 5.—Relation of Corrosion Rate to Carbon Dioxide Concentration.

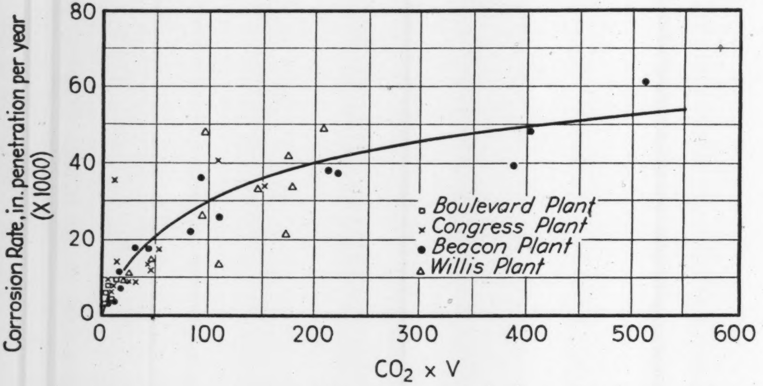

FIG. 6.—Relation of Corrosion Rate to the Product of Carbon Dioxide Concentration Multiplied by Velocity.

tion of the corrosion rate with the several variables is best shown by graphical means.

There are three variables which may affect the corrosion rate: namely, the amounts of oxygen and carbon dioxide dissolved in the condensate and the rate of flow. The amounts of oxygen and carbon dioxide dissolved in the condensate were somewhat less than the amounts entrained in the steam.

A study of the values in Table II shows that there is little agreement between the oxygen content of the condensate and the rate of corrosion. This becomes even more evident when we examine Fig. 4, in which the corrosion rate is plotted against the oxygen content. It therefore appears that, although oxygen is undoubtedly a factor in heating system corrosion, its effect, at least under the conditions of these tests, is minor as compared with the effect of the amounts of carbon dioxide present.

Figure 5 shows the corrosion rate plotted against the carbon dioxide content. The points are rather widely scattered, but they show generally an increasing corrosion rate with increasing carbon dioxide content.

In the attempt to take into account the rate of flow, Fig. 6 was constructed, in which the corrosion rate is plotted against the product of the carbon dioxide content and the velocity of the condensate in the 1-in. pipe (last column, Table II). The points in Fig. 6 show a definite trend, sufficient to warrant drawing a curve. When the same points are plotted to logarithmic coordinates (Fig. 7), they fall with fair consistency about a straight line.

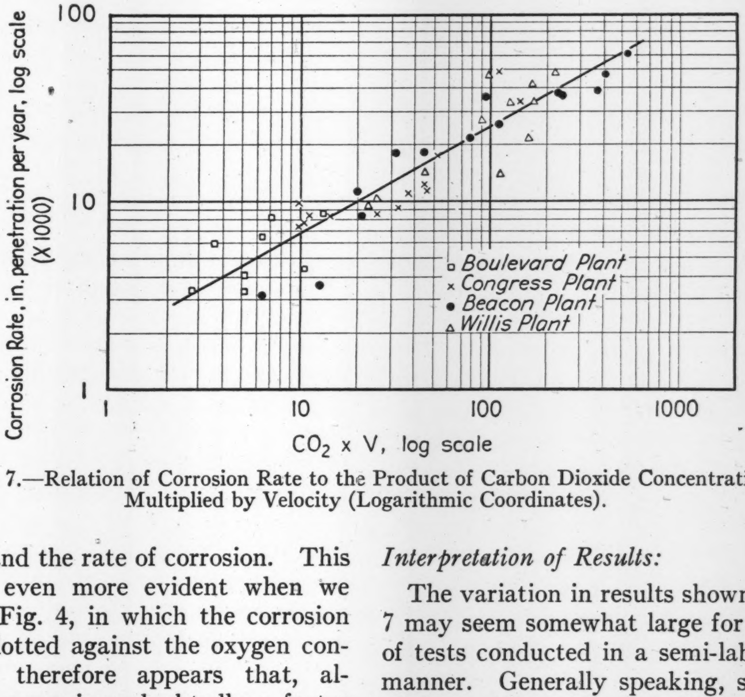

FIG. 7.—Relation of Corrosion Rate to the Product of Carbon Dioxide Concentration Multiplied by Velocity (Logarithmic Coordinates).

Interpretation of Results:

The variation in results shown in Fig. 7 may seem somewhat large for a series of tests conducted in a semi-laboratory manner. Generally speaking, such variations might be due either to the failure to recognize and measure some additional variable or to inaccuracies in the measurement of known variables. In this case, one very probable reason for the spread of the results is the fluctuation of the quantity of carbon dioxide dissolved in the condensate. The condensate analyses shown in Table II are the averages of the results of about five spot samples taken during each test period. With fluctuating conditions, the accuracy of spot sampling is not good unless a large number of samples are taken. The less-than-perfect accuracy of the evolution method of analysis for carbon dioxide, particularly at low concentrations, is also a factor.

The exact physical effect of the rate of flow is not clear. The empirical use of the quantity $CO_2 \times V$ in Figs. 6 and 7 served to bring the plotted points into line and indicates that there was some functional relationship between rate of flow and the corrosion of the tester. However, the tests were made in only one size of pipe, and there is no certainty that tests with other pipe sizes would show a similar relationship. Neither is there any evidence that the corrosion rate of a pipe would have a relationship to the quantity $CO_2 \times V$ similar to the relationship found for the tester. The effect of the velocity of flow in these tests was unmistakable, but there is not enough information to draw general conclusions about it. It appears, however, that it is a factor that must not be neglected in corrosion tests.

All of the tests were purposely made with a nearly constant steam pressure in the supply line to the condenser, and the condensate temperatures, therefore, did not vary greatly. Practical experience with corrosion in heating systems seems to indicate that corrosion increases with temperature, as might be expected. Temperature is a factor in corrosion measurement that must not be lost sight of.

The results of these tests, together with other evidence, have led to the conclusion by the Detroit Edison Co. that the carbon dioxide in the steam supplied from its heating plants is a much more important factor than the oxygen, and changes are being made in feedwater treatment to reduce the carbon dioxide content of the steam to less than 5 ppm.

Advantages and Limitations of the N.D.H.A. Tester:

The test results presented and the experience gained in the tests afford a basis for a critical consideration of the N.D.H.A. tester and of testers of that general type.

For the purpose for which it was intended, the N.D.H.A. tester has served very well indeed. From a mechanical standpoint, it is an extremely valuable device because it can be inserted in the piping system without much cost or inconvenience. As an approximate method of comparing corrosiveness in different heating systems, it is probably fairly reliable, but more study is needed of the significance of the rate-of-flow effect before exact comparisons are justified.

If the rate-of-flow effect is not limited to this particular design of tester but is a general factor in corrosion, then any corrosion test must take it into account. Furthermore, the possible differences in the effect of rate of flow on the corrosion of the tester and on the corrosion of the pipe surfaces must be considered.

Another important question regarding this general type of corrosion test is whether the loss in weight of a corrosion specimen is a proper index of the corrosiveness of an environment. What one is ordinarily interested in when considering the corrosiveness of waters is the effect of such waters upon pipes and other containers. The life of a pipe is usually considered to be ended either when the wall becomes thinned below permissible limits or when it has been perforated by pitting. The loss in weight of a corrosion specimen might possibly be translated into the probable effect on the life of a pipe, if the corrosion is reasonably uniform, but it cannot be done if the corrosion effect is one of pronounced pitting. There is much justification for the argument that to determine the corrosion effect on a pipe, a piece of the pipe itself should be used as the corrosion specimen and that the number and depth of pits should be taken into account rather than merely the loss in weight. The mechanical difficulties in using specimens of pipe, especially in large sizes, are of course the objections to that method.

With proper attention to such matters as frequency of sampling and analysis, control and measurement of rate of flow, and careful standardization of method, a device such as the N.D.H.A. tester could probably be used to measure the corrosiveness of natural waters, providing loss of weight were acceptable as the criterion.

SUMMARY

The N.D.H.A. corrosion tester has proven to be a satisfactory device for the purpose for which it was designed. It has the advantages of simplicity and ease of insertion in the pipes of a heating system.

When used in a semi-laboratory test of the corrosiveness of condensate by the Detroit Edison Co., the tester gave reasonably consistent results when the pronounced effect of rate of flow of the condensate over the tester was taken into account.

Rate of flow must be considered as one of the variables when using the N.D.H.A. tester, and this may be true with any similar device.

A corrosion test which uses loss of weight of a specimen as the index of corrosiveness may not give a true picture of the practical effect of corrosion, because the corrosion may take the form of pronounced pitting.

Other factors such as the frequency of sampling of the water and the accuracy of the analyses should be taken into account when a corrosion test is used to compare the corrosiveness of different waters.

Acknowledgments:

The corrosion tests were conducted by the staff of the Central Heating Department of the Detroit Edison Co. The chemical work was done by L. F. Collins.

APPENDIX

Derivation of the formula for calculating corrosion rate:

$$R = \frac{D_1}{2T}\left(\frac{W_1 - W_2}{W_1 + \sqrt{W_1 W_2}}\right)$$

Let

D_1 = initial diameter in inches,
D_2 = final diameter in inches,
L = length in inches,
W_1 = initial weight in pounds,
W_2 = final weight in pounds,
d = density in pounds per cubic inch,
T = time of exposure in years, and
R = corrosion rate in inches penetration per year.

$$\text{The initial area} = \pi D_1 L$$

$$= \left(\frac{\pi D_1^2 L}{4}\right)\frac{4}{D_1} = \frac{4}{D_1} \times \frac{W_1}{d}$$

$$\text{Final area} = \frac{4}{D_2}\frac{W_2}{d}$$

Now $$\frac{D_1^2}{D_2^2} = \frac{W_1}{W_2}$$

$$D_2 = D_1\sqrt{\frac{W_2}{W_1}}$$

$$\text{and the final area} = \frac{4W_2}{d} \times \frac{1}{D_1\sqrt{\frac{W_2}{W_1}}}$$

$$= \frac{4}{dD_1}\sqrt{W_1 W_2}$$

The corrosion rate is assumed to be constant and the average area of exposure is therefore the average of the initial and final areas, or

$$\text{Average area} = \frac{\frac{4}{D_1} \times \frac{W_1}{d} + \frac{4}{D_1 d}\sqrt{W_1 W_2}}{2}$$

$$= \frac{2}{D_1 d}(W_1 + \sqrt{W_1 W_2})$$

$$R \times T \times \text{area} \times d = W_1 - W_2$$

$$R = \frac{W_1 - W_2}{dT}$$

$$\times \frac{D_1 d}{2(W_1 + \sqrt{W_1 W_2})}$$

$$R = \frac{D_1}{2T}\left(\frac{W_1 - W_2}{W_1 + \sqrt{W_1 W_2}}\right)$$

DISCUSSION

MR. THOMAS J. FINNEGAN[1] (*presented in written form*).—Mr. Walker states in his paper that the N.D.H.A. corrosion tester has been regarded as a means of measuring the corrosiveness of the environment rather than as an indication of the life of equipment. This is an important point which cannot be emphasized too strongly. If we allow two different solutions to flow over corrosion testers installed in similar pipes, it is reasonable to believe that the solution which produced the greater corrosion of the tester is the more corrosive solution. The actual life of piping depends upon whether the corrosion is uniform or in the form of pits, that is, upon the original condition of the pipe and its subsequent behavior when exposed to the solution. In general, the pipe life will be less when exposed to the more corrosive solution, but its actual length cannot be determined by a tester of this type.

Attempts to express the results of the weight loss of specimens in terms of life of pipe may be greatly influenced by the use of the unit "inches per year penetration" to express corrosion rate. Actually this unit is more or less of a fiction and merely takes the small amount of weight loss during the period of test and relates it to the original weight by means of the assumption that all the corrosion is uniform. If the testers were always made of wire 0.05 in. in diameter and the tests were all of 28 days duration, or 0.0767 yr., and if in addition the difference between W_1 and W_2 is small enough so that $\sqrt{W_1W_2}$ is essentially equal to W_1, the formula for R reduces to

$$0.163\left(\frac{W_1 - W_2}{W_1}\right).$$

The constant serves only to place the quantity on an inches per year basis, and, if so desired, it could be dropped and the corrosion rate expressed as the ratio of the weight loss to the original weight. This would reduce the likelihood of making erroneous applications of the results of a test to the life of piping, as well as making the final calculation very easy.

The effect of the rate of flow might be explained by the nature of the corroding liquid used in these tests. Steam condensate when first formed is quite unbuffered, and, depending upon its pH value, is more or less corrosive to the metal near the point of condensation. At the same time, it becomes less corrosive as the liquid remains in contact with the metal as, for instance, in flowing through a condensate return pipe. This has been shown experimentally by Mr. L. F. Collins of Mr. Walker's staff.

If a strong mineral acid were used in the tests, the rate of flow might be unimportant, but when a delicately balanced material like steam condensate is used it might be that the time of contact of the liquid and the metal is quite important in determining whether one obtains poor spacing of the points, as in Fig. 5, or fair spacing, as in Fig. 6. It is true that the apparatus of Fig. 3 is mainly of non-ferrous metal, but this would influence the magnitude of the velocity effect rather than eliminate it.

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[1] Research Associate, Consolidated Edison Company of New York, Inc., New York, N. Y.

If the apparatus were all of iron or steel the effect of velocity might be much more pronounced.

The experiments using the apparatus of Fig. 3 show that the tester functions to the satisfaction of the experimenter in the determination of the corrosiveness of condensed steam. It would seem, however, that a great advantage of the tester is that it can be installed in an actual operating system and is, therefore, a useful tool for field tests. The classification of values of $R \times 1000$ as negligible, mild, and active, were obtained in such a field study, and are, therefore, more convincing than if they were found in laboratory tests. This tester need not be restricted to work on steam condensate; with more study it might be extended to many practical corrosion problems in industry. The tester can be considered as capable of producing a short-time corrosion test without the risks attendant to so-called accelerated tests.

MR. J. C. WARNER[2] (*presented in written form*).—The data presented in this paper show that results of some significance can be obtained by the use of the "corrosion tester" and the author has judiciously considered the limitations as well as the possibilities of this method of testing the corrosiveness of a water.

The results of this investigation, like many which are undertaken to obtain an approximate practical result in a short time, could be used as the basis for planning an investigation which should yield less ambiguous results of more general applicability. For example, it would be interesting to expose testers with O_2 and CO_2 concentrations, rate of flow, temperature, pipe diameter, etc., held constant for the duration of the test and to subject a number of testers to apparently identical conditions to determine how many apparently identical tests would be required to obtain the same average corrosion rate, within 5 per cent or 10 per cent, time after time. Such an investigation would involve the application of the statistical methods which have been so successfully applied to many corrosion studies.[3]

Once results of known reproducibility have been obtained over a considerable range in the values of the important variables, it might be possible, by the use of dimensional analysis, to find ways of translating the results obtained in laboratory or semilaboratory tests to practical large scale equipment.

It is much easier, however, to talk about these comprehensive careful investigations than it is to find time to do them, and one cannot blame the busy technical man for dropping a problem when he has obtained an approximate answer.

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[2] Professor of Chemistry, Carnegie Institute of Technology, Pittsburgh, Pa.

[3] U. R. Evans, "Metallic Corrosion, Passivity and Protection," pp. 665–667, Edward Arnold and Co., London (1937).

SYMPOSIUM ON SPECTROGRAPHIC ANALYSIS

VARIABLES *VERSUS* CONSTANTS IN EMISSION SPECTROGRAPHY

BY THOMAS A. WRIGHT[1]

Quantitative spectrography is an art built on the foundations of a science. Usually the art has preceded the science. Therein, perhaps, lies the reason for certain errors to be met with on occasion—errors which may not always be recognized in that they arise from a false sense of security or too great an optimism. The seemingly simple is after all not so simple.

The present symposium is definitely related to a further symposium being held at the 1940 annual meeting, namely, on Tools of Analytical Chemistry, for the spectrograph is very definitely a tool, and a newer one of the analytical chemist.

The two sessions of the Symposium on Spectrographic Analysis, however, have been designed for a particular audience—one composed not only of our guest chemists from the American Chemical Society, but also of guest speakers in the persons of the chairmen, vice-chairmen, or representatives of certain of the Society's standing committees. These men are essentially metallurgical engineers. They represent those phases of A.S.T.M. work which at the moment are most closely concerned with present applications of spectrography. Being engineers by training, they have an attitude and approach with which the chemist-spectrographer or physicist-spectrographer should become familiar. But it is also true that they should have a better acquaintance with the spectrographer and the spectrographer with the engineer. That is, the engineer should know better what can be expected from the spectrographer.

It is quite evident that by and large a primary goal for Committee E-2 on Spectrographic Analysis is the development of Go-No-Go methods for the checking of engineering compositional specifications and that it is a main excuse for its existence. Naturally the strictly quantitative or actual factual type result is often required too, but it is subordinate to the Go-No-Go just as the simple qualitative (or the more complex study of compositional differences) is in turn subordinate to the straight quantitative with which the analytical chemist supplies the engineer.

It is true too that during the development, improvement, or revision of a specification, one or the other type of report may acquire a temporary and greater importance, but by and large the Go-No-Go could be our main objective. Must we then concentrate on the development of Go-No-Go's to the exclusion of the others? The answer is obviously "no" for several reasons, among which may be mentioned the following:

1. The Go-No-Go method is peculiarly applicable to mass production engineering and to mass production spectrography.

2. As such it concentrates on one—or in any event on only a relatively few specific elements whereby one may readily ascertain—within limits of a

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[1] Technical Director and Secretary, Lucius Pitkin, Inc., New York, N. Y.

commercial reproducibility or approach to accuracy—whether or not the composition of an alloy or other raw material, intermediate, accessory or final product is in accordance with plant requirements or A.S.T.M. (or other) specifications.

3. But we have just suggested that a Go-No-Go method may go far beyond checking the final engineering product specification. Indeed the materials that together compose the final metal or alloy or other product may themselves, and often do, come under a specification. This then is a subject of peculiar interest to Subcommittee III on Quantitative Methods and Applications and this subcommittee has specifically been brought into this symposium through its chairman.

4. We have still not explored the field. We still must consider the strictly quantitative determination of one or more elements. There are innumerable applications of this of course but for illustration we may for the moment confine ourselves to one. Reference is made to those cases involving remelt metal wherein contamination or concentration of an impurity or loss of an important but minor ratio element(s) may have occurred. For example, in remelting die-cast scrap, lead may have built up over the 0.007 per cent maximum, or magnesium may have been burnt out below an 0.02 or 0.03 per cent minimum. Obviously one must ascertain the actual quantity so that proper adjustment with new metal may be made. The approach to accuracy is most important at the borderline minimum and maximum. These are practical questions but the answer may and often should involve very scientific means—in other words, Fundamental Methods and Technique. This then is obviously the function of Subcommittee II whose chairman is also a selected speaker.

As for the subjects for discussion, we are almost immediately faced with the complexity of emission spectrography technique. Not that we are unaware that absorption plays its part in the design of the instrument or in plate or film registration, but absorption spectrography as an art is not our concern at the moment. Rather is it the limitations of emission spectrography. We read and hear much of its possibilities, but little of its limitations, aside from what we hear in the lobby or corridors. We should hear more, for only by open self-criticism and open admission are we going to progress as we should. The time is past when spectrography needs lyrical salesmen. There is a large field for improvement and extension, but the latter should come from the former. Let us then choose as our main subject these limitations.

As for what they are, we must define spectrography as we know it. To define it we must certainly know the various steps comprised in the making of a spectrographic analysis. Apparently we must digress (perhaps it is not a digression) and consider the term "spectrographic analysis." Now obviously neither a chemical analysis (whether the conventional macro or the newer micro) nor a spectrographic quantitative analysis is necessarily accurate. After 35 yr. experience one may say, in fact one must say, they usually are not truly accurate. One can go further and say they usually need not be. This is probably heretical.

What is necessary, however, is that the approach to accuracy be known, or, as some would put it, the degree of accuracy, or the degree of precision. Let us also for the sake of clarity use, instead of precision, the word "reproducibility," for we spectrographers are mightily concerned with this matter of reproducibility, particularly as it is related to three items, which are excitation (especially in the solution method), photographic plate registration, and plate development in all methods.

For convenience then, let us recognize this approach to accuracy by three terms which show in order this approach. These terms are in everyday use in our own laboratory for our own and our clients' edification. They are, respectively, Determinations, Estimates, and Guesses. Now it is quite obvious that experience in spectrography is just as favorable a factor as it is in any other science, art, or trade. Such being the case, it is just as obvious that the guess of a skilled worker in a given field may be as nearly accurate where minor amounts of element X are concerned as the estimate of another worker not so familiar with the element or the matrix. Obviously, too, the determination of one not familiar with the method or technique may be no nearer the truth than the estimate of another. We ought to recognize these distinctions although not necessarily the particular phraseology given here. They are legitimate distinctions. There is a need for each of these types. They merely bridge in one way the abrupt line between quantitative and qualitative which exists only in the dictionary. Put one way, a high-grade qualitative analysis is a low-grade quantitative analysis. The point is to recognize and make the distinction. The less we mislead ourselves, the less we mislead instead of help and guide those who use our results.

With that background as to the degree of accuracy desired or to be attained, we can now take up the various steps in this quantitative, quasi-, or quali-quantitative, or plain qualitative analysis. These are in the main as follows, assuming that only one type of arc or spark excitation is available alone or together:

1. Sample
  A. Choice of
  B. Preparation of
    1. For direct arcing of
      a. Solid material
      b. Solution technique (See sec. *2*)
      c. Dried salts technique
    2. For sparking of
      a. Solid material
      b. Solution technique (See sec. *2*)
      c. Dried salts technique
  C. Decomposition of
  D. Concentration of
  E. Isolation of selected elements for purposes of
    1. Concentration
    2. Freeing from interfering matrix or allied elements or compounds.
  F. Selection of preferred weight
  G. If a non-conductor, amount and method of incorporating graphite or other conductor (reducer)

2. Solution Method
  Where solution method is in view considerations such as:
  A. Size of both upper and lower electrodes
  B. Preburning lower
  C. Pretreating to insure an impervious crater or
  D. Impregnating as method of charging sample
  E. Impregnating as source of internal standard
  F. Use of an absorbent pellet to receive sample solution.
  G. Slow *versus* rapid drying—apparatus and technique therefor

3. Spectral Range or Position(s)
  If large Littrow prism or grating, choice or selection is dependent upon elements studied

4. Excitation
  A. Choice of method of excitation
    1. Arc Types, and for example:
      a. Whether lower is to be the positive or negative
    2. Spark Types, and for example:
      a. Application of cathode ray oscillograph

- B. Excitation period
  - 1. Arc
    - *a.* Stated period (partial)
    - *b.* Complete burn.
  - 2. Spark
    - *a.* Fixed sample
    - *b.* Moving sample
- C. Excitation potential
  - 1. Arc
  - 2. Spark
- D. Method of starting arc
  - 1. Initial potential
  - 2. Major potential

5. Choice of Electrodes—Arc Method

- A. Self electrodes
- B. Graphitized carbon
  - 1. Regular purity
  - 2. High purity
  - 3. Special forms
  - 4. Size of crater (width, depth and capacity)
  - 5. Thickness of crater wall
  - 6. Form of crater
- C. Carbon
  (See also sections *5* B and *2*)
- D. Copper
- E. Silver
- F. Method of preparation of A, B, C, D, or E.
- G. Method of charging selected material.

6. Photography

- A. Slit width
- B. Choice of plates for
  - 1. Regular or medium U.V. range (for example, 2500–3600 Å)
  - 2. Lower U.V. end
  - 3. Red end
- C. Film—as indicated
- D. Registration period
  - 1. *a.* Without sector (same as excitation period)
    - *b.* With sector
  - 2. Qualitative only
  - 3. Quantitative methods
    - *a.* Comparative standard
    - *b.* Internal standard
    - *c.* Line (and band) spectra
    - *d.* Log sector
    - *e.* Step sector
- E. Methods of development
  - 1. Effect of plate or film storage conditions
  - 2. Developing temperature, time and concentration
  - 3. Regular developing solutions
  - 4. Special developing solutions
  - 5. Tank *versus* tray developing
  - 6. Automatic *versus* hand development
  - 7. Mass production methods
  - 8. Special plate methods
  - 9. Washing
  - 10. Fixing
  - 11. Rapid drying *versus* slow drying

7. Examination or Measurement

- A. Weighting of irregular spectra (light and dark)
- B. Background corrections
- C. Visual (one *versus* multiple observers)
  - 1. Direct examination of plate
    - *a.* Apparatus for
    - *b.* Choice of magnification
  - 2. Projection
    - *a.* Using standards on same plates (or film)
    - *b.* Using reference or standard plates (or film)
- D. Micro-densitometers
  - 1. Direct reading
  - 2. Recording

8. Standards

- A. Standard alloy forms
  - 1. Chill-cast rods and vertical ingots
    - *a.* For spark use
    - *b.* For low amperage arc use
  - 2. For arc use
    - *a.* Sawings
    - *b.* Raspings
    - *c.* Millings
    - *d.* Drillings
    - *e.* Powders
      - *1.* Conducting (thermal and electrical)
      - *2.* Non-conducting (thermal and electrical)
      - *3.* High *versus* low melting point
- B. Solution standards
  - 1. Synthetic alloy formulas
    - *a.* Soluble readily
    - *b.* Soluble with difficulty
    - *c.* Containing readily volatile or precipitable constituents
  - 2. For use as comparative standards—routine
  - 3. For use in calibrating a method

9. *Sensitivity*
   - A. As governed by major elements or general matrix composition
   - B. As governed by allied minor associates
   - C. As governed by weight of sample employed

D. As governed by acidic radical	Solution methods
E. As governed by alkalinity	

   - F. Choice of appropriate wave-lengths for a given concentration
   - G. Other factors inferred previously

10. *Presentation, Interpretation and Use of Results*

Preliminary Use	A. As a guide to further spectrographic technique	
	B. As an aid to the analytical chemist	
	C. As a guide to the engineer or other technologist	
Research Use	D. In developing a method	By the analyst or the physicist
	E. In proving a method	
Routine Use	F. By the producer of the material	
	G. By the user of the material	
	H. By the layman	

Almost any one of a number of subjects touched upon in this survey could be the subject of a symposium in itself. We have selected, however, for discussion at the morning session "Accuracy Requirements *versus* Reproducibility Possibilities." Can spectrographers consistently give the metallurgist or engineer results within 2 or 5 or 10 per cent of the truth? Can the engineer consistently ask us for 2 per cent when 5 per cent or 10 per cent would do?

In the afternoon session we shall consider the "Presentation of Results and Their Interpretation." For example: How are we going to report minor amounts? Which terms should we use and when, as for example: per cent, parts per thousand, parts per million, milligrams per liter, milligrams per c.c. or per ml., or gamma? Or when we give the engineer a quasi-quantitative report what does he think we spectroscopists think we mean when we put down, major, minor, appreciable, low, strong, weak, intermediate, trace or spectrographic trace? What other terms can be used when we have no suitable previous experience with that particular matrix or base material, no suitable standard or reference?

Perhaps we shall have the answer to some of these questions as a result of the symposium and thus be encouraged to go further and shortly engage to attack the problem of nomenclature.

[The Symposium on Spectrographic Analysis was an interchange of views between the engineer or metallurgist who develops and uses metallurgical specifications, and the analytical spectrographer. Prepared discussion was solicited from a number of selected individuals, which was supplemented by oral discussion. This discussion will be published in the ASTM BULLETIN.—ED.]

SUMMARY OF PROCEEDINGS OF THE DETROIT SPRING MEETING—SYMPOSIUM ON NEW MATERIALS IN TRANSPORTATION

The eleventh spring meeting of the Society was held in Detroit, Mich., Wednesday, March 6, 1940, in conjunction with the Spring Group Meetings of the A.S.T.M. Committees.

The morning and afternoon sessions of the meeting comprised a technical program—a Symposium on New Materials in Transportation. This symposium was under the auspices of the Detroit District Committee. The committee immediately in charge of the technical program consisted of the following:

Carl E. Heussner (*Chairman*), Materials Engineer, Chrysler Corp.
W. H. Graves (*Acting Chairman*), Chief Metallurgist, Packard Motor Car Co.
Peter Altman, Aeronautical Dept., University of Detroit.
S. D. Heron, Research Engineer, Ethyl Gasoline Corp.
J. W. Kennedy, Engineer, Service Bureau, Huron Portland Cement Co.
J. L. McCloud, Metallurgical Chemist, Ford Motor Co.

The purpose of this symposium was not only to emphasize some of the major current developments but also to indicate trends which may be of increasing significance in the future. The symposium consisted of the following seven papers:

"Exhaust Valve Materials for Internal-Combustion Engines," by S. D. Heron, Director of Aeronautical Research, Ethyl Gasoline Corp., O. E. Harder, and M. R. Nestor, Assistant Director, and Research Engineer, respectively, Battelle Memorial Institute.

"Selection and Application of Automotive Steels," by A. L. Boegehold, Head, Metallurgy Dept., Research Laboratories Division, General Motors Corp., W. H. Graves, Chief Metallurgist, Packard Motor Car Co., and E. W. Upham, Chief Metallurgist, Chrysler Corp.

"Rubber of Tomorrow," by Sidney M. Cadwell, Director, Automotive Development, United States Rubber Co.

"Advances in the Uses of Concrete in Transportation," by Miles D. Catton, Civil Engineer, Development Dept., Portland Cement Assn.

"Recent Developments in the Use of Asphalt for Transportation Purposes," by Bernard E. Gray, Chief Engineer, The Asphalt Institute.

"Trends in the Properties of Volatile Liquid Fuels," by D. P. Barnard and A. H. Fox, Associate Director of Research, and Research Engineer, respectively, Standard Oil Co. (Indiana).

"Developments in Lubrication," by J. P. Stewart, R. C. Moran, and O. M. Reiff, Head, Automotive Section, Supervising Chemist, and Research Chemist, respectively, Research and Development Division, General Laboratories, Socony-Vacuum Oil Co., Inc.

The chairmen of the sessions were as follows:

Morning Session.—W. M. Barr, Chief Chemical and Metallurgical Engineer, Union Pacific Railroad Co.

Afternoon Session.—J. L. McCloud, Metallurgical Chemist, Ford Motor Co.

The papers in this symposium elicited a considerable amount of discussion which contributed greatly to the information contained in the papers. The papers, complete with discussion, have been published by the Society as a separate volume entitled, "Symposium on New Materials in Transportation."

The evening session—the Spring Meeting Dinner—featured an address on "The Antiquity of Things New in Transportation," by J. S. Worley, Professor of Transportation Engineering and Curator of the Library of Transportation, University of Michigan.

T. A. Boyd, Head Fuel Dept., General Motors Research Laboratories, General Motors Corp., was toastmaster at the dinner.

There were approximately 250 members and guests in attendance at the sessions.

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